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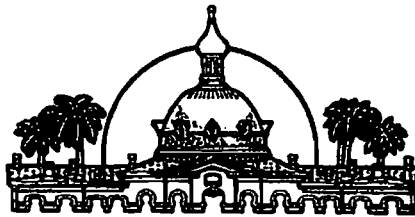
# Findings and Recommendations

## Continuous Monitoring of Mercury Emissions at a Municipal Waste Combustor

January 2011



*Report*



**Hillsborough County  
Florida**

Office of the County Administrator  
Michael S. Merrill

**BOARD OF COUNTY COMMISSIONERS**

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January 27, 2011

Scott M. Sheplak, P.E.  
Department of Environmental Protection  
Division of Air Resources Management  
Mail Station #5505  
2600 Blair Stone Road  
Tallahassee, FL 32399-2400  
*0570261-010-AC*  
Subject: Mercury Analyzer Report

**RECEIVED**

JAN 28 2011

BUREAU OF  
AIR REGULATION

Dear Mr. Sheplak,

This letter is a follow up to our meeting on October 22, 2010 and a subsequent conference call held on November 10, 2010 to discuss selection of a mercury monitoring device for installation on Unit 4 at the County's Waste-To-Energy facility.

At the November 10<sup>th</sup> meeting, it was confirmed that Covanta would provide you with a report summarizing its experience with the mercury monitor (PS-12A mercury analyzer), currently proposed by FDEP for use at the facility. To that end, we are pleased to provide you with copies of the Covanta report.

Below is a brief summary of the points that were discussed in our earlier meeting and conference call.

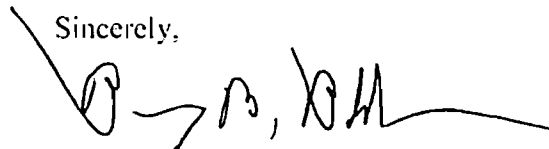
1. A system to continuously monitor mercury emissions must be installed by September 5, 2011.
2. It is the opinion of the County, Covanta, and CDM that Air Permit No. 0570261-010-AC (Permit) allows the use of either a PS-12A CEMS or an ETV-certified CEMS. While writing the permit, FDEP stated their intent to require installation of a PS-12A mercury analyzer.
3. Covanta purchased such a device from the Tekran company (a Tekran mercury analyzer) which analyzes emissions continuously and in real time. This Tekran analyzer was installed in July 2009 at the WTE facility operated by Covanta in Haverhill, MA in order for the company to gain operational experience with the device. Covanta enlisted the aid of Tekran through an annual maintenance contract to assist in the operation and maintenance of the device.

4. During this time, Covanta experienced great difficulty in keeping the Tekran analyzer on line for more than 3 weeks at a time. A component of the analyzer, known as a gold trap, rapidly deteriorated and had to be replaced on a frequent basis at a cost of approximately \$1,500 per occurrence. Covanta believes the nature of flue gas from a waste to energy (WTE) facility is the root cause of deterioration. This resulted in very low operational availability of the Tekran analyzer at the Haverhill facility.
5. In contrast, according to Covanta and CDM research, an ETV-certified CEMS, known as a sorbent trap, has proven to be much more reliable in operational applications. The ETV-certified CEMS (also known as a PS-12B CEMS) is less expensive to install and much less costly to maintain than the PS-12A analyzer. Therefore, if forced to utilize the FDEP proposed device, the County would be forced to pay higher installation and O&M costs.
6. If the alternate device (PS-12B CEMS) were approved for use to continuously monitor mercury emissions, the County could use a portion of the cost savings to fund a Mercury Bounty Program where citizens would be encouraged to drop off items containing mercury and receive some type of reward, such as a gift certificate to local businesses. This would assist in achieving the important goal of removing mercury from the environment.
7. Covanta was to prepare a detailed report of the Tekran analyzer performance at Haverhill and submit to all parties in January 2011.
8. A meeting between all parties would be scheduled shortly after Covanta submits the report.

Based upon our review of this report, the County is of the opinion that the ETV-certified/PS-12B CEMS is the best choice for the continuous monitoring of mercury emissions. It complies with USEPA rules and the current PSD Permit language for unit No. 4 at the County's WTE facility. This device will provide a higher availability than the PS-12A analyzer at a far lower cost; and conserves County financial resources that could be used for a Mercury Bounty Program to accomplish what all parties desire: removal of mercury from the environment.

Hillsborough County requests a meeting within the next three weeks with the appropriate FDEP staff to discuss this report and to finalize a course of action.

Sincerely,



Barry M. Boldissar, Division Director  
Solid Waste Management Division,  
Public Utilities Department

Attachment: Mercury Analyzer Report

Cc: Michael S. Merrill, County Administrator, Hillsborough County  
Paul J. Vanderploog, Director, Public Utilities, Hillsborough County  
Nate Johnson, Hillsborough County  
Jason Gorrie, Covanta Energy  
Glenn Hoag, Covanta Energy  
Dan Strobridge, CDM  
Bill Crellin, CDM

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**ATTACHMENTS**

- Attachment 1: Tekran Analyzer Daily Checklists
- Attachment 2: Performance Specification 12B for Mercury CEMS
- Attachment 3: ETV Certificate and Report for HG-324K Mercury Monitor
- Attachment 4: Analyzer Data and Trends

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AIR REGULATION**

## Purpose of Report

This Report has been prepared by Covanta Energy ("Covanta") to document the findings related to the installation and operation of a Tekran® Series 3300 Continuous Mercury Analyzer (hereinafter, the "Tekran analyzer") at the Haverhill Resource Recovery Facility located in Haverhill, Massachusetts. In July 2009, Covanta, at its sole expense, purchased and installed the Tekran analyzer to obtain operational experience with Mercury Continuous Emissions Monitoring Systems ("CEMS") on a Municipal Waste Combustor. It is specifically noted that there are numerous analyzers used to continuously monitor mercury (or "Hg") emissions on coal-fired utility boilers throughout the United States. However, because the flue gas composition of coal-fired utility boilers is different from the flue gas composition of MWCs, Covanta decided to install the Tekran analyzer to verify vendor claims that the technology was equally applicable to MWCs as it was to coal-fired utility boilers. To Covanta's knowledge, the Tekran analyzer at the Haverhill facility is the only fully-operational Hg CEMS installed on a Municipal Waste Combustor ("MWC") in the United States. This Report summarizes Covanta's operational experience with the Tekran Series 3300 CEMS at the Haverhill Resource Recovery Facility and provides Conclusions and Recommendations on the best course of action to continuously monitor mercury emissions from Unit #4 at the Hillsborough County Resource Recovery Facility.

## Description of the System

The Tekran analyzer is comprised of several interconnected systems including a sample probe, a sample conditioner, and a mercury detector. The sample probe is a dilution-based system that continuously draws stack gas and eliminates particulates with an inertial separator. The sample conditioner separates the mercury into elemental ( $\text{Hg}^0$ ) and ionic ( $\text{Hg}^{2+}$ ) species and removes  $\text{SO}_2$  and other gases that can cause interference in the detector. The mercury detector uses pure gold preconcentration and cold vapor atomic fluorescence to measure the amount of mercury present in the sample. Figure 1 depicts a simplified process diagram of the entire system.

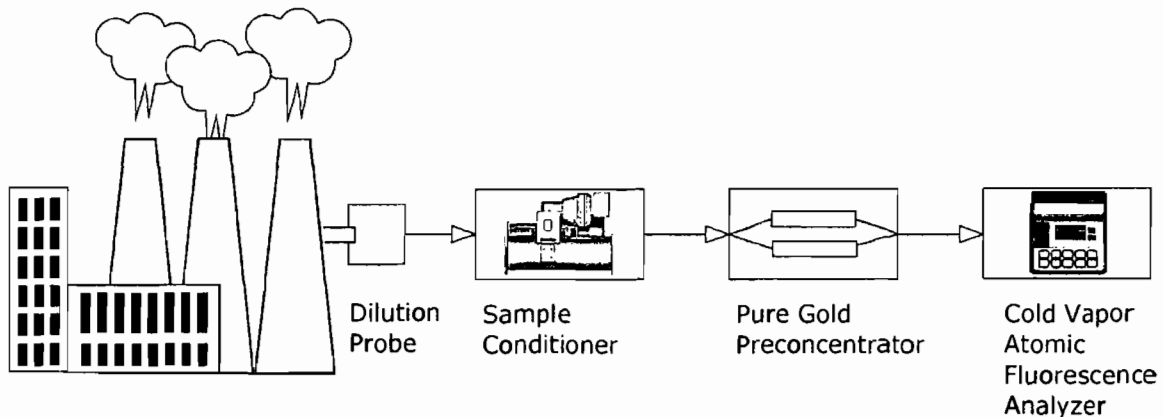


Figure 1  
Tekran® Hg CEMS

## Summary of Monitor Performance

The Tekran analyzer has operated nearly continuously since July 2009. However, the data collected by the system is frequently qualified as bad because internally-measured parameters within the instrument fall out of specification. The most common of these out-of-specification parameters are related to the pure gold preconcentration equipment within the mercury detector, as described below.

### *Gold Trap*

An integral component of the mercury detector is the pure gold preconcentration equipment, hereinafter referred to as the "gold trap". The gold trap consists of a pair of canisters that are assembled in parallel and contain gold. Mercury has a high affinity for gold. The purpose of the gold trap is to temporarily "trap" the mercury on the gold before it is sent to the cold vapor atomic fluorescence unit for quantification<sup>1</sup>. Conditioned sample gas is directed to one of the gold traps in the parallel assembly allowing mercury to accumulate on the gold. While one trap is accumulating mercury, the other trap in the parallel assembly is being desorbed with argon gas and directed to the detector. A solenoid valve automatically cycles the sample gas between the two canisters every 2 ½ minutes.

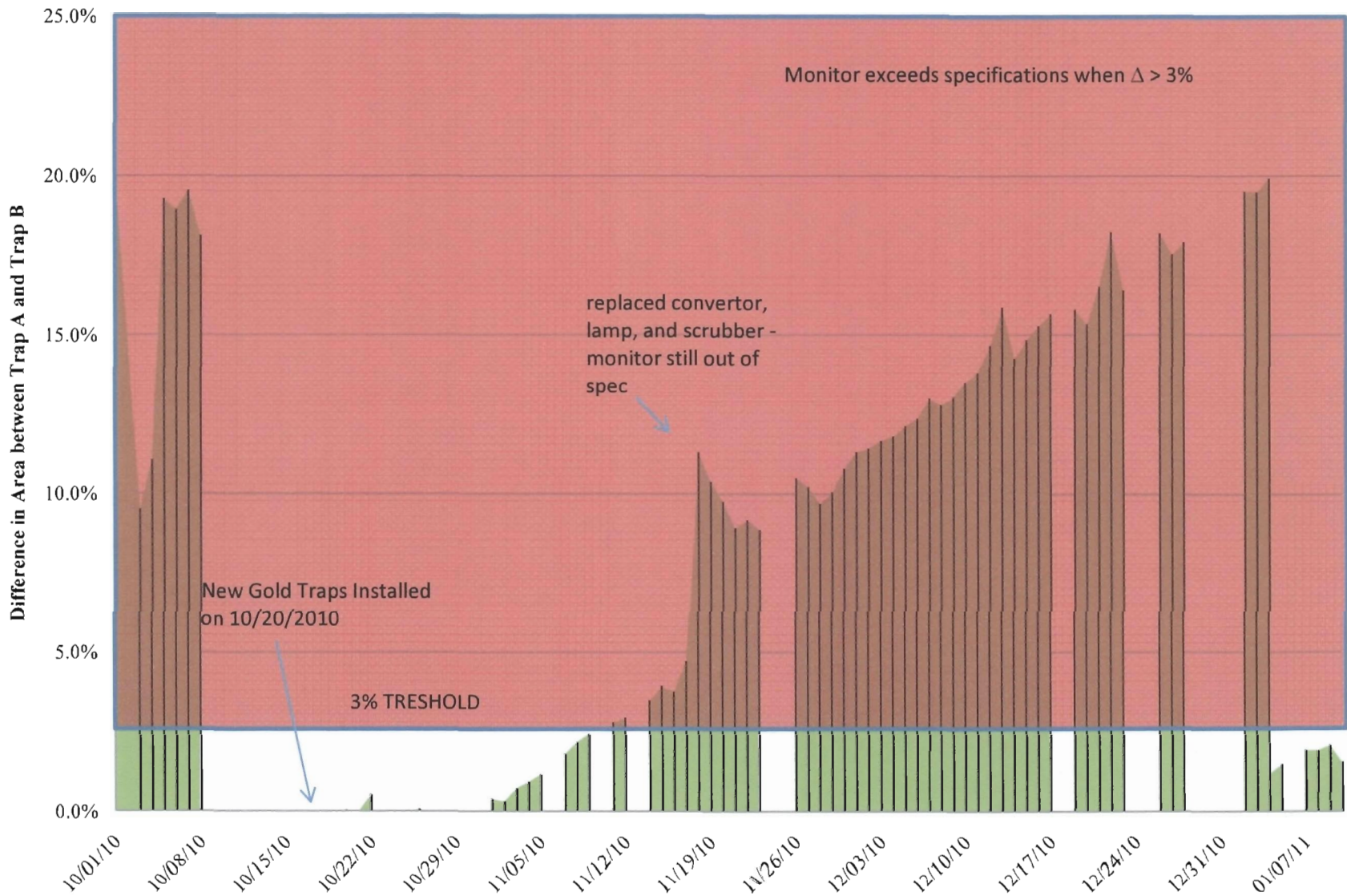
The Tekran instrument internally diagnoses the integrity of the gold trap by periodically measuring the area of gold available upon which mercury accumulates. The difference between the available areas within the two canisters is calculated and serves as an indication of the integrity of the overall gold trap. If this difference exceeds 3%, the instrument is considered to be operating out of specification.

**Figure 2** presents the measured delta between the two canisters for the period of October 1, 2010 until January 10, 2011. Of particular note on Figure 2 is the period between October 20<sup>th</sup> and November 12<sup>th</sup>. Following a series of gold trap failures over the summer of 2010, the analyzer was shipped back to Tekran for diagnosis and service. On October 19<sup>th</sup>, Tekran technicians re-installed the analyzer at Haverhill and assisted in re-startup of the system. Following this intensive two-week factory service by the analyzer manufacturer, the analyzer was again exceeding the 3% delta specification by November 12<sup>th</sup>, a period of only 23 days. While this 23 day runtime following the factory service is somewhat better than previous runtimes (which on average have been approximately 2 weeks), it is still a relatively short period for an analyzer that is required to continuously monitor mercury emissions.

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<sup>1</sup> Tekran states that this pure gold preconcentration step reduces variable interferences that are observed in other technologies used to continuously measure mercury.

**Figure 2**  
**Monitor Data Quality**



## **Efforts to Improve Monitor Reliability**

Gold trap degradation and failure has been the principal cause of monitor unavailability to date. Covanta and Tekran have undertaken a number of different actions to understand the root cause of the gold trap failure and to extend the life of the expensive component, including a visit to the Brayton Point Power Station in Somerset, Massachusetts where the Tekran analyzer operates with little gold trap degradation. As of this writing, the cause of the gold trap degradation is believed to be a function of the flue gas characteristics<sup>2</sup>. The analyzer O&M procedures being undertaken at Haverhill are similar to the O&M procedures employed at Brayton Point (Attachment 1 provides a copy of the Daily Checklist utilized at Haverhill.) The significant difference between the fuels (coal at Brayton Point, municipal solid waste at Haverhill) leads Covanta to believe that the gold trap degradation observed at Haverhill may be caused by trace pollutants which are not present in the flue gas of a typical coal fired boiler.

In order to keep the Tekran analyzer functioning, Covanta has purchased and replaced the gold trap assembly on the following dates since June 1, 2010: June 29, 2010; July 22, 2010; August 9, 2010; August 16, 2010; October 20, 2010; and, January 2, 2011. In each instance, the trap area gradually decayed and the observed delta of the trap area exceeded the 3% specification within a 2 to 3 week period. The analyzer was shipped back to the Tekran warehouse on October 8, 2010. This was in response to an observation from Tekran technicians that the flow setting on the analyzer may be too high, causing the rapid degradation of the gold traps. Tekran modified the flow setting, installed new gold traps, and assisted Covanta personnel in restarting the analyzer on October 21, 2010. Even with this manufacturer modification, the gold traps were again operating above the 3% delta specification in less than 3 weeks (see Figure 2).

The latest system modification undertaken by Tekran and Covanta entailed installing a new critical orifice in the dilution probe to increase the dilution ratio from 30:1 to 50:1. The new critical orifice was installed on December 22, 2010, new gold traps were also installed and the analyzer was placed back into service on January 2, 2011. As of the writing of this Report, the traps have not significantly degraded. We are hopeful that the increased dilution ratio will prolong the service life of the gold traps, but this potential solution remains unproven. Further, the modified dilution ratio will do nothing to remove the presence of chlorine that was identified by Tekran when they analyzed contaminants on a failed gold trap assembly.

## **Conclusions**

Fundamentally, the Tekran analyzer is capable of continuously measuring mercury emissions. However, the experience at Haverhill indicates that the flue gas characteristics of a municipal waste combustor degrade the integrity of the gold traps at a much faster rate than has been observed at coal-fired boiler installations. To date, Tekran and Covanta have been unable to determine the exact cause of the gold

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<sup>2</sup> On 09/15/2010, Tekran reported to Covanta via an email transmission that an analysis of a failed gold trap found chlorine as a suspect contaminant



trap degradation. Several attempts to regenerate the gold traps have failed. The only solution identified to date is to replace them at a cost of approximately \$1,500 each time.

Covanta and Hillsborough County acknowledge the Department's stated desire to "drive the current state of the technology" through permit conditions, and appreciate the 2 year grace period afforded by Condition No. 33.b. of Permit No. PSD-369B to achieve 95% monitor availability. However, the experience at Haverhill for the past 1 ½ years indicates that the actions for restoring the monitor to service following gold trap degradation requires replacing the gold trap component at a frequency of approximately every two weeks. This frequency of gold trap replacement will cost Hillsborough County \$40,000 annually in parts alone, not counting additional labor to install the traps and uncertainty in the future price of gold.

Covanta has diligently undertaken efforts to improve the performance and availability of the Tekran analyzer, including the purchase of Tekran's "Premier Annual Hg CEMS Maintenance Agreement" at a cost of approximately \$40,000 (which does not include the cost of gold trap replacement). Similarly, Tekran has provided invaluable assistance in diagnosing problems and restoring the analyzer to service. Nonetheless, the gold traps continue to fail, necessitating a costly replacement to achieve monitor availability.

## Discussion

Covanta and Hillsborough County have discussed with the Department the viability of utilizing a sorbent trap based system to continuously monitor mercury emissions for Unit 4 at the Hillsborough County Resource Recovery Facility. This method of continuous mercury emissions monitoring is accepted by the USEPA, has been allowed by the FDEP at other facilities<sup>3</sup>, and is fully capable of continuously measuring total mercury emissions to the environment<sup>4</sup>. Further, the cost savings that can be achieved by utilizing a sorbent trap based system to continuously monitor mercury emissions can be used by Hillsborough County to implement mercury-in-the-wastestream reduction programs that will lead to *actual* mercury emissions reductions.

As previously discussed with the Department on a number of different occasions, the USEPA has promulgated Performance Specification 12B for Mercury CEMS, which relies on sorbent trap technology (see Attachment 2). In addition to the promulgated Performance Specification, the sorbent trap based method for continuously monitoring mercury emissions has passed verification tests conducted under the auspices of the USEPA's Environmental Technology Verification (ETV) program (see Attachment 3). In turn, the USEPA has recently implemented emissions limiting standards for several industries that

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<sup>3</sup> See Specific Condition No. 17 of Permit No. PSD-FI-375 issued to Seminole Generating Station No. 3

<sup>4</sup> Until real-time operational experience is obtained, it remains uncertain what monitor availability can be achieved for a sorbent-trap based CEMS. However, discussions with other sources utilizing sorbent trap-based CEMS suggests that 85% availability is readily achievable.

require the continuous monitoring of mercury emissions and allow for the use of PS-12B/ETV-certified sorbent trap based CEMS. Indeed, the FDEP even participated in some of these USEPA rulemaking efforts by providing comments in support of the requirement for continuously monitoring mercury emissions, yet the FDEP stated no objection to the use of sorbent trap based CEMS to satisfy such requirements.

The FDEP's stated concern that a sorbent trap based system will not allow facility operator's to observe emission spikes "instantaneously" is irrelevant. Instantaneous feedback to the control room will NOT allow operators to adjust control equipment to minimize emissions. Mercury spikes are brief in duration (see Attachment 4). Any reaction by control room operators to instantaneous, real-time data from a mercury analyzer would be futile. Once a mercury-containing device has been combusted, the liberated mercury disrupts the equilibrium reaction kinetics occurring across the filtercake (lime, carbon, and ash) of the baghouse, resulting in Hg "breakthrough" (i.e. an observed emissions spike). After-the-fact injection of additional carbon will do nothing to reduce Hg emissions and will only serve to waste reagent.

It is firmly established that bioaccumulation is the threat presented by mercury in the environment. A PS-12B CEMS (sorbent trap based) is just as capable as a PS-12A CEMS (analyzer based) in recording emissions that occur in a sporadic manner (i.e., the intermittent spikes that cumulatively contribute to mercury loading in the environment will be reflected in the total mass loading recorded.) As such, higher data availability obtained over long periods should be the goal of all parties concerned. The benefit of the Tekran analyzer's ability to integrate data over shorter time periods is diminished by its inability to measure mercury emissions "at all" when it is operating out of specification, as documented above.

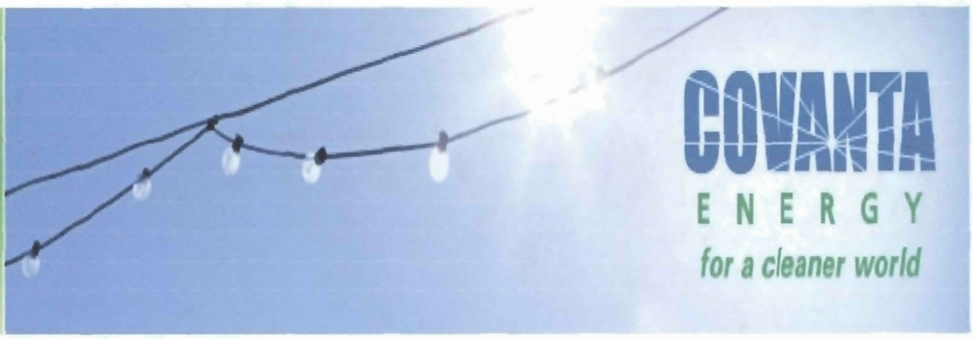
Lastly, the Department recently issued a permit to the Palm Beach County Solid Waste Authority for the Palm Beach Renewable Energy Facility No. 2 that states that the mercury CEMS is to be used to demonstrate compliance with a 12 month rolling monthly average (presumably in recognition of the long-term bioaccumulative threat that mercury emissions present.) Instantaneous data availability is not necessary to demonstrate compliance with a 12 month rolling monthly average<sup>5</sup>.

Most importantly, emissions monitoring technologies do nothing to reduce mercury emissions. The goal of the Department, Covanta, and Hillsborough County should be to remove mercury from the wastestream. To that end, Covanta has successfully participated in mercury bounty programs at communities throughout the United States. These programs are designed to provide citizens and businesses incentives to exchange mercury containing devices for gift cards to local restaurants and establishments. To maximize the effectiveness of these programs, it is important to advertize their existence and to fund the incentive – both of which compete for limited local funds. If Hillsborough County is required to install a monitoring technology that will cost in excess of \$100,000 per year to maintain, they will be less able to implement programs such as the mercury bounty program described above which will actually reduce mercury emissions. The cost savings that can be achieved by installing a

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<sup>5</sup> PSD-FL-369B does not specify an averaging period

PS-12B CEMS (sorbent trap based) in lieu of a PS-12A CEMS (analyzer based) can be used in part to maximize mercury reduction programs without any reduction in mercury emissions monitoring capability.



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# ATTACHMENT 1

## Tekran Analyzer Daily Checklists



### Series 3300 Mercury CMM Daily Checklist Log

Rev 1.06 October 08, 2009

Typical Settings		2537 Analyzer					3320 Sample Conditioner						1304 Air Panel			3310 Calibrator			3340 Probe Controller				3342 Probe		System	
Date	Time	Argon Delivery Pressure in Range	[BL] Baseline Voltage in Range	Trap 'A' Baseline Deviation in Range	Trap 'B' Baseline Deviation in Range	[VOL] Sample Volume Normal	[HEATED LINE] Unleakal Zone Temp Normal	Converter Temp in Range	Chiller Temp in Range	[OVEN] Hot Box Temp Normal	Sufficient Water Supply	Bypass Flow in Range	Inlet Air Supply Normal	Purge Air Supply Normal	Zero Air Supply Normal	Preheater Temp	Hg Source Temp	MFC Plate Temp	Bypass Air Pressure in Range	Dilution Air Pressure in Range	Bypass Flow in Range	Orifice Vacuum Normal	Sample Cell Temp in Range	Singer Temp in Range	System Controller, No alarms	Initials
12/20/10	7:28	51	.113	.550	.099	1.21	178	775	5.1	180	NO	✓	90	90	90	85.0	15.0	40.0	9.4	50.0	✓	✓	210	194	✓	F.P.
12/21/10	7:30	50	.112	.103	.113	1.20	178	775	4.9	150	Y	325	88	86	88	87.0	15.0	40.0	9.5	50.0	✓	-19.91	210	199	✓	JK
12/22/10	7:30	50	.113	.142	.110	1.31	178	775	5.0	180	Y	325	87	87	87	85.0	15.0	40.0	9.5	50.0	✓	-19.95	210	207	✓	JK
12/23/10	7:30	50	.112	.119	.125	1.20	178	775	4.9	180	Y	325	90	90	90	85.0	15.0	40.0	9.8	50.0	✓	-20.63	210	198	✓	JK

Weekly Checks		2537 Analyzer			3320 Sample Conditioner			1306 Water Sys			Argon			A Trap		B Trap		CF	CF	
Date	Time	Every 48 Hours	Off	Less Than 3% Relative	less than 1/3 Full	Moving Bubbles in Drains	H <sub>2</sub> O Present	Less Than 5.9 g/lime	Total Dissolved Solids	Total Dissolved Solids	Pressure	Pressure	In Service	Initials	Dilution ratio	Inst. Det. Adj %				
12-20-10	7:32	✓	✓	14.19	✓	✓	✓	.09	133	007	700	1900	1	F.P.	31.780574	12.15	998,516	872192	1.093	1.120
															31.780574	14.20	1001501	859334	1.141	1.140
															31.740574	15.05	1013375	960892	1.108	1.121
															50.5000	15.34	1013220	857819	1.108	1.121

↳ system back to normal, water flow through system.

\* NEW CRITICAL ORIFICE installed 50:1

Cartridge Deviation = ((Larger Trap Area - Smaller Trap Area) / Larger Trap Area) x 100%

12-13-10  
to  
12-14-10  
TEKRAV

Series 3300 Mercury CMM  
Daily Checklist Log

Rev 1.06 October 08, 2009

Typical Settings		2537 Analyzer					3320 Sample Conditioner					1304 Air Panel			3310 Calibrator			3340 Probe Controller				3342 Probe		System		
Date	Time	Argon Delivery Pressure in Range	[BL] Baseline Voltage In Range	Trap "A" Baseline Deviation in Range	Trap "B" Baseline Deviation in Range	[VOL] Sample Volume Normal	[HEATED LINE] Umbilical Zone Temp Normal	Converter Temp in Range	Chiller Temp in Range	[OVEN] Hot Box Temp Normal	Bullient Water Supply	Bypass Flow in Range	Inlet Air Supply Normal	Purge Air Supply Normal	Zero Air Supply Normal	Preheater Temp	Hg Source Temp	MFC Plate Temp	ByPass Air Pressure In Range	Dilution Air Pressure in Range	ByPass Flow in Range	Office Vacuum Normal	Sample Cell Temp in Range	Singer Temp in Range	System Controller, No alarms	Initials
12/13/10	10:18	50	.117	.125	.150	1.21	178	775	5.1	180	✓	✓	91	91	91	85.0	15.0	40.0	9.4	50.0	✓	✓	210	208	✓	F.P.
12/14/10	7:18	50	.114	.034	.032	1.21	178	775	5.0	180	✓	✓	89	89	89	85.0	15.11	40.0	9.4	49.9	✓	✓	210	205	X Yes	F.P.
12/15/10	6:22	50	.112	.032	.025	1.21	178	775	5.0	180	✓	✓	87	87	87	85.0	15.0	40.0	9.5	50.0	✓	✓	210	199	✓	F.P.
12/16/10	10:20	50	.112	.029	.029	1.21	178	775	5.0	180	✓	✓	88	88	88	85.0	15.0	40.0	9.5	50.0	✓	-	210	193	✓	F.P.
12/17/10	7:13	50	.112	.035	.039	1.21	178	775	5.0	181	✓	✓	90	90	90	85.0	15.0	40.0	9.4	50.0	✓	✓	210	199	✓	F.P.

Weekly Checks		2537 Analyzer			3320 Sample Conditioner			1304 Water Sys			Argon			Dilution ratio	Instr. Det. Adj %	A Trap	B Trap	A CF	B CF	
Date	Time	Every 48 Hours	Off	Less Than 3% Relative	Less than 1/3 Full	Moving Bubbles in Drain	H <sub>2</sub> O Present	Less Than 8.0 μS/cm	Total Dissolved Solids	Total Dissolved Solids	Pressure	Pressure	In Service	Initials						
12-13-10	10:22	✓	✓	14.07	✓	✓	✓	.08	097	001	1600	300	1	F.P.	31.780574	12.44	2,022,044	1,770,402	1.120	1.122
2-14-10	05:16	Alarm - Calibration Recovery out of Range - cleared																		
2-18-10		Alarm - Lamp voltage out of Range - adjuster Trim pot - cleared																		
															31.780574	13.26	1,914,697	1,660,852	1.150	1.141
															31.780574	13.56	1,920,685	1,660,273	1.085	1.084
															31.780574	13.72	1,910,440	1,648,392	1.085	1.084

Cartridge Deviation = ((Larger Trap Area - Smaller Trap Area) / Larger Trap Area) x 100%

Series 3300 Mercury CMM  
Daily Checklist Log

Rev 1.06 October 08, 2009

Typical Settings		2637 Analyzer					3320 Sample Conditioner					1304 Air Panel			3310 Calibrator			3340 Probe Controller				3342 Probe		System		
Date	Time	Argon Delivery Pressure in Range	[BL] Baseline Voltage in Range	Trap "A" Baseline Deviation in Range	Trap "B" Baseline Deviation in Range	[VOL] Sample Volume Normal	[HEATED LINE] Umbilical Zone Temp Normal	Converter Temp in Range	Chiller Temp in Range	[OVEN] Hot Box Temp Normal	Sufficient Water Supply	Bypass Flow in Range	Inlet Air Supply Normal	Purge Air Supply Normal	Zero Air Supply Normal	Pre-heat Temp	Hg Source Temp	MFC Plate Temp	Bypass Air Pressure in Range	Dilution Air Pressure in Range	Bypass Flow in Range	Orifice Vacuum Normal	Sample Cell Temp in Range	Blinger Temp in Range	System Controller, No alarms	Initials
11/29/10	7:12	151	.116	.026	.035	1.21	178	775	5.1	181	✓	✓	88	88	88	85.0	15.0	40.0	9.5	50.0	✓	✓	210	198	✓	F.P.
11/30/10	7:12	51	.111	.036	.028	1.21	178	776	5.1	181	✓	✓	89	89	89	85.1	15.0	40.0	9.4	50.0	✓	✓	210	193	✓	F.P.
12/1/10	6:47	50	.110	.027	.029	1.21	178	775	4.9	180	✓	✓	90	90	90	85.0	14.9	40.0	9.4	50.0	✓	✓	210	210	✓	F.P.
12/2/10	6:50	50	.111	.026	.027	1.21	178	775	4.9	180	✓	✓	89	89	89	85.0	14.9	40.0	9.4	50.0	✓	✓	210	193	✓	F.P.
12/3/10	6:33	50	.112	.025	.024	1.21	178	775	4.9	180	✓	✓	90	90	90	85.0	14.9	40.0	9.4	50.0	✓	✓	210	193	✓	F.P.
12/4/10	6:47		.111	.037	.032	1.21	178	775	5.0	180	✓	✓	88	88	88	85.0	14.9	40.0	9.5	50.0	✓	✓	210	209	✓	F.P.

Weekly Checks		2637 Analyzer					3320 Sample Conditioner			1306 Water Sys			Argon			A Trap		B Trap		CF	
Date	Time	Instrument Detector Adjustment Current	Lamp Light Off	Cartridge Deviation Normal	Sufficient Waste Water Storage	Scrubbers Draining Properly	Water Drain Trap Sealed	Running Conductivity Normal	TD6 In	TD6 Out	Cylinder 1	Cylinder 2	Cylinder 1 (Left) or 2 (Right)	Dilution ratio	Instr. Det. Adj %	A Trap	B Trap	CF	CF		
11/29	7:20	✓	✓	9.16	✓	✓	✓	.06	107	66	1760	1350	2	F.P.	31,780,574	9.16	2,010,224	1,826,052	1.099	1.099	
															31,780,574	9.20	2,016,239	1,830,831	1.099	1.099	
															31,780,574	10.33	1,931,267	1,731,831	1.099	1.099	
															31,780,574	10.04	1,912,691	1,720,664	1.099	1.099	
															31,780,574	10.44	1,945,026	1,741,957	1.099	1.099	

Cartridge Deviation = ((Larger Trap Area - Smaller Trap Area) / Larger Trap Area) x 100%

31,780,574 10.13 1,900,298 1,696,467 1.099 1.099

11-22-10  
 11-27-10 T. Rillo  
 TEKRAN

Series 3300 Mercury CMM  
 Daily Checklist Log

Rev 1.06 October 08, 2009

Typical Settings		2837 Analyzer					3320 Sample Conditioner						1304 Air Panel			3310 Calibrator			3340 Probe Controller				3342 Probe		System	
Date	Time	Argon Delivery Pressure in Range	[BL] Baseline Voltage in Range	Trap "A" Baseline Deviation in Range	Trap "B" Baseline Deviation in Range	[VOL] Sample Volume Normal	[HEATED LINE] Urbitical Zone Temp Normal	Converter Temp in Range	Chiller Temp in Range	[OVEN] Hot Box Temp Normal	Sufficient Water Supply	Bypass Flow in Range	Min 95 psi	Min 90 psi	Min 90 psi	At Setpoint	At Setpoint	At Setpoint	9 to 15 psi	35 to 60 psi	1 to 1.5 l/min	Less Than -15.8" Hg	180° C to 228° C	180° C to 228° C	Status Page	Initiate
11/20/10	8:31	50	.114	.026	.037	1.21	178	775	5.1	180	✓	✓	89	89	89	85.0	15.0	40.0	9.4	50.0	✓	✓	210	201	✓	F.P.
11/22/10	6:43	52	.111	.036	.026	1.21	178	775	4.9	180	✓	✓	88	88	88	85.0	14.9	40.0	9.4	50.0	✓	✓	210	207	✓	F.P.
11/23/10	7:05	51	.114	.026	.032	1.21	178	775	4.9	179	✓	✓	88	88	88	85.0	14.9	40.0	9.4	50.0	✓	20.20	210	201	✓	F.P.
11/24/10	8:05	51	.114	.025	.031	1.21	178	775	4.9	180	✓	✓	89	89	89	85.0	15.0	40.0	9.5	50.0	✓	✓	210	205	✓	F.P.

Weekly Checks		2837 Analyzer			3320 Sample Conditioner			1304 Water Sys			Argon			Diffusion ratio	Instr. Det. Adj %	A Trap	B Trap	A CF	B CF	
Date	Time	Every 48 Hours	Oil	Less Than 2% Relative	less than 1/3 full	Moving Bubbles in Drains	H <sub>2</sub> O Present	Less Than 0.5 p.p.m. Residue	Total Dissolved Solids	Total Dissolved Solids	Pressure	Pressure	In Service	Initiate						
11-22	6:46	✓	✓	8.29	✓	✓	✓	0.06	112	000	1780	1680	2	F.P.	31.780574	8.78	1,912,443	1,744,568	1.067	1.084
																1.				

Cartridge Deviation = (Larger Trap Area - Smaller Trap Area) / Larger Trap Area x 100%





### Series 3300 Mercury CMM Daily Checklist Log

Rev 1.06 October 08, 2009

Typical Settings		2637 Analyzer					3320 Sample Conditioner						1304 Air Panel			3310 Calibrator			3340 Probe Controller				3342 Probe		System	
Date	Time	Argon Delivery Pressure in Range	[BL] Baseline Voltage in Range	Trap 'A' Baseline Deviation in Range	Trap 'B' Baseline Deviation in Range	[VOL] Sample Volume Normal	[HEATED LINE] Umbilical Zone Temp Normal	Converter Temp in Range	Chiller Temp in Range	[OVEN] Hot Box Temp Normal	Sufficient Water Supply	Bypass Flow in Range	Inlet Air Supply Normal	Purge Air Supply Normal	Zero Air Supply Normal	Preheater Temp	Hg Source Temp	MFC Plate Temp	Bypass Air Pressure in Range	Dilution Air Pressure in Range	By-Pass Flow in Range	Orifice Vacuum Normal	Sample Cell Temp in Range	Stinger Temp in Range	System Controller, No alarms	Initials
11/15/10	6:55	50	.108	.024	.036	1.21	178	775	5.1	180	✓	✓	90	90	90	85.0	15.0	40.0	9.4	50.0	✓	✓	210	196	✓	F.P.
11/16/10	6:21	50	.108	.031	.031	1.21	178	775	5.1	180	✓	✓	90	90	90	85.0	15.1	40.0	9.4	50.0	✓	✓	210	209	✓	F.P.
11/17/10	9:36	50	.108	.036	.028	1.21	178	775	5.1	180	✓	✓	88	88	88	84.9	15.0	39.9	9.4	50.0	✓	✓	210	209	✓	F.P.
11/18/10	8:15	50	.111	.037	.034	1.21	178	776	5.0	180	✓	✓	93	93	93	85.1	15.0	40.0	9.4	50.0	✓	✓	210	205	✓	F.P.
11/19/10	9:46	49.5	.113	.026	.044	1.21	178	775	4.9	180	✓	✓	88	88	88	45.0	15.0	40.0	9.5	50.05	✓	20.3	210	204	✓	JK
11/20/10	8:31	50	.114	.026	.037	1.21	178	775	5.1	180	✓	✓	89	89	89	85.0	15.0	40.0	9.4	50.0	✓	✓	210	201	✓	F.P.

Weekly Checks		2637 Analyzer					3320 Sample Conditioner					1304 Water Sys			Argon			Dilution ratio		A Trap		B Trap		A CF		B CF		
Date	Time	Every 48 Hours	Off	Less Than 5% Relative	Less than 1/3 full	Moving Bubbles in Drains	H <sub>2</sub> O Present	Less Than 6.9 μ Siemens	Total Dissolved Solids	Total Dissolved Solids	Pressure	Pressure	In Service	Initials			lost. Det. Adj %											
11-15-10	6:58	✓	✓	3.93	✓	✓	✓	0.07	113	0	700	1700	1	F.P.	31.780574	3.93	4.13	1,468,242	1,528,231	1.072	1.051							

Cartridge Deviation = ((Larger Trap Area - Smaller Trap Area) / Larger Trap Area) x 100%



### Series 3300 Mercury CMM Daily Checklist Log

Rev 1.06 October 08, 2009

Typical Settings		2637 Analyzer					3320 Sample Conditioner						1304 Air Panel			3310 Calibrator			3340 Probe Controller				3342 Probe		System	
Date	Time	Argon Delivery Pressure in Range	[BL] Baseline Voltage in Range	Trap "A" Baseline Deviation in Range	Trap "B" Baseline Deviation in Range	[VOL.] Sample Volume Normal	[HEATED LINE] Urbi/Cal Zone Temp Normal	Converter Temp in Range	Chiller Temp in Range	[OVEN] Hot Box Temp Normal	Sufficient Water Supply	Bypass Flow in Range	Inlet Air Supply Normal	Purge Air Supply Normal	Zero Air Supply Normal	Pre-Heater Temp	Hg Source Temp	MFC Plate Temp	Bypass Air Pressure in Range	Dilution Air Pressure in Range	Bypass Flow in Range	Onice Vacuum Normal	Sample Cell Temp in Range	Stinger Temp in Range	System Controller, No alarms	Initials
11/8/10	7:08	51	.108	.025	.032	1.21	180	775	5.0	180	✓	✓	86	86	86	85.0	15.0	40.0	9.5	50.0	✓	✓	210	206	✓	F.P.
11/9/10	7:02	50	.108	.026	.038	1.21	178	775	5.1	180	✓	✓	90	90	90	84.7	15.0	40.0	9.4	50.0	✓	✓	209	204	✓	F.P.
11/10/10	7:05	50	.108	.031	.023	1.21	178	775	5.1	180	✓	✓	90	90	90	85.0	15.0	40.0	9.4	50.0	✓	✓	210	209	✓	F.P.
11/11/10	8:02	50	.108	.021	.025	1.21	178	775	5.0	180	✓	✓	90	90	90	84.7	15.0	40.0	9.4	50.0	✓	✓	209	195	✓	F.P.
11/12/10	6:51	50	.109	.024	.030	1.21	178	775	5.1	179	✓	✓	89	89	89	84.7	14.9	39.9	9.5	50.0	✓	✓	210	209	✓	F.P.

Weekly Checks		2637 Analyzer			3320 Sample Conditioner			1304 Water Sys			Argon			Dilution ratio	Inst. Det. Adj. %	A Trap	B Trap	A CF	B CF	
Date	Time	Every 48 Hours	Off	Less Than 3% Relative	Less than 1/3 full	Moving Bubbles in Drains	H <sub>2</sub> O Present	Less Than 6.8 μS/cm	Total Dissolved Solids	Total Dissolved Solids	Pressure	Pressure	In Service	Initials						
11-8-10	7:17	✓	✓	2.33	✓	✓	✓	0.06	0	0	1200	1700	1	K.P.	31,780,574	2.33	1,513,947	1,550,083	1.052	1.031

Cartridge Deviation = ((Larger Trap Area - Smaller Trap Area) / Larger Trap Area) x 100%

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### Series 3300 Mercury CMM Daily Checklist Log

Rev 1.06 October 08, 2009

Typical Settings		2637 Analyzer					3320 Sample Conditioner						1304 Air Panel			3310 Calibrator			3340 Probe Controller				3342 Probe		System	
		40-60 psi	0.05 to 0.19	0 to 0.1	0 to 0.1	1.20	180° C	700° C to 800° C	3° C to 5° C	180° C	No loss than 1/3 full	300 to 350 ml/min	Min 95 psi	Min 90 psi	Min 90 psi	Air Setpoint	Air Setpoint	Air Setpoint	9 to 18 psi	35 to 60 psi	1 to 1.5 l/min	Less Than -15.5" Hg	180° C to 220° C	180° C to 220° C	Status Page	
Date	Time	Argon Delivery Pressure in Range	[BC] Baseline Voltage in Range	Trap "A" Baseline Deviation in Range	Trap "B" Baseline Deviation in Range	[VOL] Sample Volume Normal	[HEATED LINE] Umbilical Zone Temp Normal	Converter Temp in Range	Chiller Temp in Range	[OVEN] Hot Box Temp Normal	Surfactant Water Supply	Bypass Flow in Range	Inlet Air Supply Normal	Purge Air Supply Normal	Zero Air Supply Normal	Preheater Temp	Hg Source Temp	MFC Plate Temp	ByPass Air Pressure in Range	Dilution Air Pressure in Range	ByPass Flow in Range	Office Vacuum Normal	Sample Cell Temp in Range	Stinger Temp in Range	System Controller, No alarms	Initials
11/1/10	6:55	51	.110	.024	.025	1.21	178	775	5.0	180	✓	✓	95	95	95	85.0	14.9	40.0	9.4	50.0	✓	20.3	210	201	✓	F.P.
11/2/10	6:47	50	.109	.020	.026	1.21	178	775	5.1	180	✓	✓	95	95	95	85.0	14.9	40.0	9.3	50.0	✓	20.43	210	193	✓	F.P.
11/3/10	8:12	51	.110	.025	.032	1.21	178	776	4.9	180	✓	✓	95	95	95	85.0	15.0	40.0	9.4	50.0	✓	20.11	210	208	✓	F.P.
11/4/10	11:34	50	.109	.030	.038	1.21	178	775	5.1	180	✓	✓	88	88	88	85.0	15.0	40.0	9.4	50.0	✓	20.09	210	209	✓	F.P.
11/5/10	11:33	50	.107	.020	.037	1.21	178	775	5.0	180	✓	✓	90	90	90	85.0	15.0	40.0	9.4	50.0	✓	19.78	210	197	✓	F.P.

Weekly Checks		2637 Analyzer			3320 Sample Conditioner			1306 Water Sys			Argon			Initials	Dilution ratio	Inst. Det. Adj. %	A Trap	B Trap	A CF	B CF
		Every 48 Hours	Off	Less Than 3% Relative	less than 1/3 full	Moving Bubbles in Drains	H <sub>2</sub> O Present	Less Than 5.8 µ Siemens	Total Dissolved Solids	Total Dissolved Solids	Pressure	Pressure	In Service							
Date	Time	Instrument Detector Adjustment Current	Lamp Light Off	Cartridge Deviation Normal	Sufficient Waste Water Storage	Scrubbers Draining Properly	Water Drain Trap Sealed	Running Conductivity Normal	TDS in	TDS Out	Cylinder 1	Cylinder 2	Cylinder 1 (Left) or 2 (Right)							
11/1/10	7:44	✓	✓	0.67	✓	✓	✓	0.06	109	0	1490	1700	1	F.P.	31.780	0.67	1568380	1578906	1.016	1.005
														F.P.	31.780	0.62	1,579,012	1,588,789	1.016	1.005
														F.P.	31.780	0.81	1,597,766	1,610,802	1.016	1.005
														F.P.	31.780	1.06	1,554,822	1,571,537	1.016	1.005
														F.P.	31.780	1.12	1,533,730	1,551,175	1.016	1.005

Cartridge Deviation = ((Larger Trap Area - Smaller Trap Area) / Larger Trap Area) x 100%



### Series 3300 Mercury CMM Daily Checklist Log

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Typical Settings		2537 Analyzer					3320 Sample Conditioner						1304 Air Panel			3310 Calibrator			3340 Probe Controller				3342 Probe		System	
Date	Time	Argon Delivery Pressure in Range	[RL] Baseline Voltage in Range	Trap 'A' Baseline Deviation in Range	Trap 'B' Baseline Deviation in Range	[VOL] Sample Volume Normal	[HEATED LINE] Umbilical Zone Temp Normal	Converter Temp in Range	Chiller Temp in Range	[OVEN] Hot Box Temp Normal	Sufficient Water Supply	Bypass Flow in Range	Inlet Air Supply Normal	Purge Air Supply Normal	Zero Air Supply Normal	Preheater Temp	Hg Source Temp	MFC Plate Temp	ByPass Air Pressure in Range	Dilution Air Pressure in Range	ByPass Flow in Range	Orifice Vacuum Normal	Sample Cell Temp in Range	Singer Temp in Range	System Controller, No alarms	Initial
10/19	0621	OFF	—	—	—	—	180	775	5.0	180	✓	✓	84	84	84	250	1499	700	9.4	5000	✓	2016	210	19.4		PSS
10/19	0750	OFF	—	—	—	—	180	775	5.0	180	✓	✓	84	84	84	250	1499	900	9.4	500	✓	2016	210	19.5		PSS
10/20	0600	52	0.111	.051	.051	.00	180	776	5.0	180	✓	✓	85	85	85	249	1499	400	9.4	500	✓	2016	210	19.5		PSS
10/21	0611	52	0.104	.037	.038	.21	180	775	4.7	180	✓	✓	92	92	92	250	1494	400	9.4	49.76	✓	19.98	210	20.7	✓	PSS
10/22	0932		0.106	.039	.038	.21	180	775	4.5	180	✓	✓	93	93	93	24.7	1494	400	9.3	50.05	✓	20.11	210	19.4	✓	PSS

2537 Analyzer		3320 Sample Conditioner				1306 Water Sys			Argon			Dilution ratio		A Trap		B Trap		CF		
Date	Time	Instrument Detector Adjustment Current	Lamp Light Off	Cartridge Deviation Normal	Sufficient Waste Water Storage	Scrubbers Draining Properly	Water Drain Trap Sealed	Running Conductivity Normal	TDS In	TDS Out	Cylinder 1	Cylinder 2	Cylinder 1 (Left) or 2 (Right)	Initial	Dilution ratio	Inst. Det. Adj %	A Trap	B Trap	CF	CF
10/21	0611	✓	✓	0.17	✓	✓	✓	0.06	115	0.0	1725	275	2	PSS	32.235300	—	—	—	—	—
															32.235300	—	—	—	1.201	1.164
															32.235290	0.17	1410147	1412542	1.015	1.013
															32.235300	0.26	1395151	1398790	.999	.983

Cartridge Deviation = ((Larger Trap Area - Smaller Trap Area) / Larger Trap Area) x 100%



## Series 3300 Mercury CMM Daily Checklist Log

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Time		2637 Analyzer					3320 Sample Conditioner					1304 Air Panel			3310 Calibrator			3340 Probe Controller				3342 Probe		System	
		45-65 psi	0.02 to 0.15	0 to 0.1	0 to 0.1	1.02	120°C to 200°C	1°C to 5°C	180°C	No leaks (200-112 psi)	100 to 250 ml/min	Min ml/min	Max ml/min	Min psi	A1 Response	A2 Response	A3 Response	5 to 15 psi	35 to 60 psi	1 to 1.5 psi	Less Than -15.0" Hg	180°C to 220°C	180°C to 220°C	System Controller, No Alarms	Initials
10/12	08:16	OFF	-	-	-	180	775	5.2	180	✓	✓	93	93	93	85.0	14.99	40.0	9.3	50.00	✓	-20.11	210	193		PSS
10/13	08:19	OFF	-	-	-	180	776	5.2	180	✓	✓	90	90	70	84.9	14.9	39.9	9.4	50.00	✓	-20.27	210	202		PSS
10/14	06:44	OFF	-	-	-	180	776	5.0	180	✓	✓	92	92	92	85.0	15.0	40.0	9.4	50.00	✓	-20.27	210	193		PSS
10/15	08:23	OFF	-	-	-	180	777	5.4	180	✓	✓	92	92	92	85.0	14.99	40.0	9.3	50.00	✓	-19.57	210	209		PSS

Weekly Check		2637 Analyzer					3320 Sample Conditioner				1306 Water Sys			Argon		A Trap		B Trap		CF	
		Every 48 hours	Off	Less Than 3% Retard	Less than 1/3 full	Mounting Bubbles in Oring	H <sub>2</sub> O Present	Less Than 1.0 g Sample	Total Dissolved Solids	Total Dissolved Solids	Pressure	Pressure	In Service	Pressure	Pressure	Inst. Det. A1 %	A Trap	B Trap	CF	CF	
10/12	08:19	Instrument Detector Adjustment Current	Lamp Light Off	Cartridge Deviation Normal	Sufficient Waste Water Storage	Scrubbers Draining Property	Water Drain Trap Sealed	Running Conductivity Normal	TDS In	TDS Out	Cylinder 1	Cylinder 2	Cylinder 1 (Left) or 2 (Right)	PSS	32.235300	-	-	-	-	-	
					✓	✓	✓	0.06	115	0.0	-	-	-		32.235300	-	-	-	-		
															32.235300	-	-	-	-		
															32.235300	-	-	-	-		

Cartridge Deviation = ((Larger Trap Area - Smaller Trap Area) / Larger Trap Area) x 100%



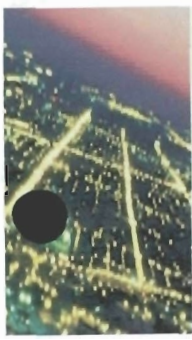
### Series 3300 Mercury CMM Daily Checklist Log

Rev 1.06 October 08, 2009

Time of Balance		2637 Analyzer					3320 Sample Conditioner					1304 Air Panel			3310 Calibrator			3340 Probe Controller			3342 Probe		System			
Date	Time	Argon Delivery Pressure in Range	[BL] Baseline Voltage in Range	Trap 'A' Baseline Deviation in Range	Trap 'B' Baseline Deviation in Range	[VOL] Sample Volume Normal	[HEATED LINE] Umbilical Zone Temp Normal	Converter Temp in Range	Chiller Temp in Range	[OVEN] Hot Box Temp Normal	Sufficient Water Supply	Bypass Flow in Range	Inlet Air Supply Normal	Purge Air Supply Normal	Zero Air Supply Normal	Preheater Temp	Hg Source Temp	MFC Plate Temp	ByPass Air Pressure in Range	Dilution Air Pressure in Range	ByPass Flow in Range	Office Vacuum Normal	Sample Cell Temp in Range	Sage Temp in Range	System Controller, No alarms	Status Page
10/4	6:45	51	0.129	0.35	0.27	0.7	180	77.5	5.0	180	✓	✓	90	90	90	850	15.01	400	9.3	50.0	✓	-20.24	210	199		PSS
10/5	8:01	51	0.144	0.55	0.24	1.24	180	77.5	5.0	180	✓	✓	89	89	89	851	15.00	399	9.3	50.0	✓	-20.29	210	204		PSS
10/6	6:51	51	0.113	0.27	0.23	1.24	180	77.5	5.0	180	✓	✓	89	89	89	850	15.00	400	9.4	50.0	✓	-20.23	210	193		PSS
10/7	11:30	50	0.146	0.23	0.33	1.24	180	77.5	5.0	18.1	✓	✓	92	92	92	84.7/14.99	399	9.3	50.0	✓	-19.26	210	209		PSS	
10/9	6:44	52	0.106	0.28	0.25	1.24	180	77.5	5.1	180	✓	✓	92	92	92	84.5/14.99	400	9.3	50.0	✓	-20.09	209	208		PSS	

2637 Analyzer		3320 Sample Conditioner			1306 Water Sys			Argon					A Trap		B Trap		CF	CF		
Steady State	Every 48 Hours	ON	Less Than 2% Relative	Less Than 1/3 full	Moving Bubbles in Drains	H <sub>2</sub> O Present	Less Than 1.0 % Leaks	Total Dissolved Solids	Total Dissolved Solids	Pressure	Pressure	In Service	Initials	Conductivity	Inst. Det. Adj %					
Date	Time	Instrument Detector Adjustment Current	Lamp Light Off	Cartridge Deviation Normal	Sufficient Waste Water Storage	Scrubbers Draining Properly	Water Drain Trap Sealed	Running Conductivity Normal	TDS In	TDS Out	Cylinder 1	Cylinder 2	Cylinder 1 (Left) or 2 (Right)							
10/4	6:51	✓	✓	NO	✓	✓	✓	0.07	117	0.0	1950	1200	Z	PSS	32.235300	15.59	959427	1136649	1.223	1.189
														PSS	32.235300	18.62	866397	1064684	1.210	1.149
															32.235300	11.14	861528	1065476	1.210	1.149
															32.235300	10.13	842462	1041749	1.229	1.182
															32.235300	19.90	845926	1053341	1.229	1.182

Cartridge Deviation = ((Larger Trap Area - Smaller Trap Area) / Larger Trap Area) x 100%



**COVANTA**  
ENERGY  
*for a cleaner world*

## ATTACHMENT 2

# Performance Specification 12B for Mercury CEMS



























## Technology Transfer Network Emission Measurement Center

<http://www.epa.gov/ttn/emc/perfspec.html>  
Last updated on Thursday, September 09, 2010









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### Promulgated Performance Specifications

From the listing below, find the file you would like to download. The available file formats are represented by icons on the right side of the listing. Click on the icon that represents the file format you wish to receive. For an explanation of file formats and their icons, see our [TTNWeb File Types](#).

File Description	Posting Date	File Types
CEMS Performance Specification 1 For Opacity	8-09-00	 
03/04/05 Memo for Opacity Monitor Manuf. May temporarily certify compliance with PS-1 following either ASTM D6216-98 or ASTM D6216-03	8-09-00	
CEMS Performance Specification 2 for SO2 and NOx	5/26/09	
CEMS Performance Specification 3 for O2 & CO2	2/2000	 
CEMS Performance Specification 4 for CO	2/2000	 
CEMS Performance Specification 4A for CO	2/2000	 
CEMS Performance Specification 4B For CO and O2 Continuous Monitoring Systems	2/2000	
CEMS Performance Specification 5 for TRS	2/2000	 
CEMS Performance Specification 6 for Flow Rate	2/2000	 
CEMS Performance Specification 7 for H2S	2/2000	 
CEMS Performance Specification 8 for VOC CEMS	2/2000	 
CEMS Performance Specification 8A for Total Hydrocarbon CEMS	9/2002	 
CEMS Performance Specification 9 for GC CEMS	2/2000	 
PS 11 for PM CEMS	01/13/04	



PS 12A for Mercury CEMS	8/9/2010	
<u>PS 12B for Mercury CEMS</u>	8/9/2010	
PS 15 for Extractive FTIR CEMS in Stationary Sources	2/2000	 
PS 16 for Predictive Emissions Monitoring Systems	4/7/2009	
40 CFR Part 60, Appendix F, Procedure 1	9/9/10	
40 CFR Part 60, Appendix F, Procedure 2	9/9/2010	
40 CFR Part 60, Appendix F, Procedure 5	8/9/2010	

**Performance Specification 12B - Specifications and Test Procedures For Monitoring Total Vapor Phase Mercury Emissions from Stationary Sources Using a Sorbent Trap Monitoring System**

1.0 Scope and Application

The purpose of Performance Specification 12B (PS 12B) is to establish performance benchmarks for, and to evaluate the acceptability of, sorbent trap monitoring systems used to monitor total vapor-phase mercury (Hg) emissions in stationary source flue gas streams. These monitoring systems involve continuous repetitive in-stack sampling using paired sorbent media traps with periodic analysis of the time-integrated samples. Persons using PS 12B should have a thorough working knowledge of Methods 1, 2, 3, 4, 5 and 30B in appendices A-1 through A-3 and A-8 to this part.

1.1 Analyte. The analyte measured by these procedures and specifications is total vapor phase Hg in the flue gas, which represents the sum of elemental Hg ( $\text{Hg}^0$ , CAS Number 7439-97-6) and gaseous forms of oxidized Hg (i.e.,  $\text{Hg}^{+2}$ ) in mass concentration units of micrograms per dry standard cubic meter ( $\mu\text{g}/\text{dscm}$ ).

1.2 Applicability.

1.2.1 These procedures are only intended for use under relatively low particulate conditions (e.g., monitoring after all pollution control devices). This specification is for evaluating the acceptability of total vapor phase Hg sorbent trap monitoring systems installed at stationary sources at the time of, or soon after, installation and whenever specified in the regulations. The Hg monitoring system must be capable of measuring the total concentration of vapor phase Hg (regardless of speciation), in units of  $\mu\text{g}/\text{dscm}$ .

1.2.2 This specification contains routine procedures and specifications designed to evaluate an installed sorbent trap monitoring system's performance over time; Procedure 5 of appendix F to this part contains additional procedures and specifications which may be required for long term operation. In addition, the source owner or operator is responsible to calibrate, maintain, and operate the monitoring system properly. The Administrator may require the owner or operator, under section 114 of the Clean Air Act, to conduct performance evaluations at other times besides the initial test to evaluate the CEMS performance. See §60.13(c) and 63.8(e)(1).

## 2.0 Principle

Known volumes of flue gas are continuously extracted from a stack or duct through paired, in-stack, pre-spiked sorbent media traps at appropriate nominal flow rates. The sorbent traps in the sampling system are periodically exchanged with new ones, prepared for analysis as needed, and analyzed by any technique that can meet the performance criteria. For quality-assurance purposes, a section of each sorbent trap is spiked with  $\text{Hg}^0$  prior to sampling. Following sampling, this section is analyzed separately and a specified minimum percentage of the spike must be recovered. Paired train sampling is required to determine method precision.

## 3.0 Definitions

3.1 *Sorbent Trap Monitoring System* means the total equipment required for the collection of gaseous Hg samples using paired three-partition sorbent traps.

3.2 *Relative Accuracy Test Procedure* means a test procedure consisting of at least nine runs, in which the accuracy of the total vapor phase Hg concentrations measured by the sorbent trap monitoring system is evaluated by comparison against concurrent measurements made with a reference method (RM). Relative accuracy tests repeated on a regular, on-going basis are referred to as relative accuracy test audits or RATAs.

3.3 *Relative Accuracy (RA)* means the absolute mean difference between the pollutant (Hg) concentrations determined by the sorbent trap monitoring system and the values determined by the reference method (RM) plus the 2.5 percent error confidence coefficient of a series of tests divided by the mean of the RM tests. Alternatively, for low concentration sources, the RA may be expressed as the absolute value of the difference between the mean sorbent trap monitoring system and RM values.

3.4 *Relative Deviation (RD)* means the absolute difference of the Hg concentration values obtained with a pair of sorbent traps divided by the sum of those concentrations, expressed as a percentage. RD is used to assess the precision of the sorbent trap monitoring system.

3.5 *Spike Recovery* means the mass of Hg recovered from the spiked trap section, expressed as a percentage of the amount spiked. Spike recovery is used to assess sample matrix interference.

#### 4.0 Interferences [Reserved]

#### 5.0 Safety

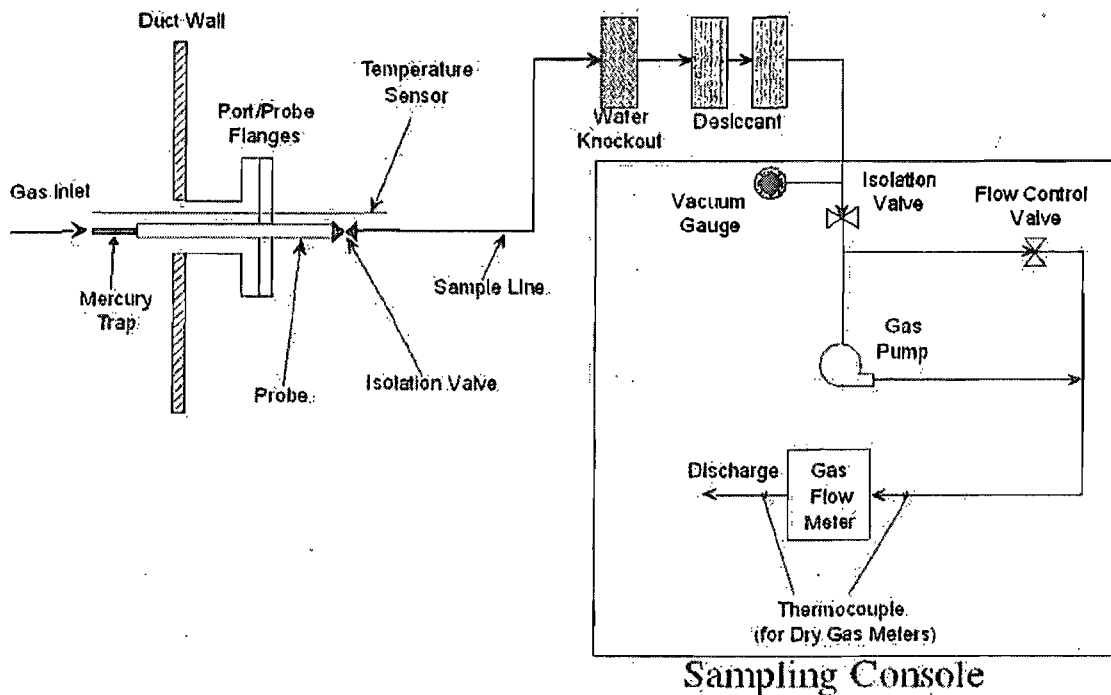
The procedures required under this performance specification may involve hazardous materials, operations, and equipment. This performance specification may not address all of the safety problems associated with these procedures. It is the responsibility of the user to establish appropriate safety and health practices and determine the applicable regulatory limitations prior to performing these procedures.

#### 6.0 Equipment and Supplies

##### 6.1 Sorbent Trap Monitoring System Equipment Specifications.

6.1.1 Monitoring System. The equipment described in Method 30B in appendix A-8 to this part must be used to continuously sample for Hg emissions, with the substitution of three-section traps in place of two-section traps, as described below. A typical sorbent trap monitoring system is shown in Figure 12B-1.

6.1.2 Three-Section Sorbent Traps. The sorbent media used to collect Hg must be configured in traps with three distinct and identical segments or sections, connected in series, to be separately analyzed. Section 1 is designated for primary capture of gaseous Hg. Section 2 is designated as a backup section for determination of vapor-phase Hg breakthrough. Section 3 is designated for quality assurance/quality control (QA/QC) purposes. Section 3 must be spiked with a known amount of gaseous Hg<sup>0</sup> prior to sampling and later analyzed to determine the spike (and hence sample) recovery efficiency.



**Figure 12B-1. Typical Sorbent Trap Monitoring System (only one trap and associated sampling system is illustrated).**

6.1.3 Gaseous  $\text{Hg}^0$  Sorbent Trap Spiking System. A known mass of gaseous  $\text{Hg}^0$  must be spiked onto section 3 of each sorbent trap prior to sampling. Any approach capable of quantitatively delivering known masses of  $\text{Hg}^0$  onto sorbent traps is acceptable. Several technologies or devices are available to meet this objective. Their practicality is a function of  $\text{Hg}$  mass spike levels. For low levels, NIST-certified or NIST-traceable gas generators or tanks may be suitable, but will likely require long preparation times. A more practical, alternative system, capable of delivering almost any mass required, employs NIST-certified or NIST-traceable  $\text{Hg}$  salt solutions (e.g.,  $\text{Hg}(\text{NO}_3)_2$ ). With this system, an aliquot of known volume and concentration is added to a reaction vessel containing a reducing agent (e.g., stannous chloride); the  $\text{Hg}$  salt solution is reduced to  $\text{Hg}^0$  and purged onto section 3 of the sorbent trap by using an impinger sparging system.

6.1.4 Sample Analysis Equipment. Any analytical system capable of quantitatively recovering and quantifying total gaseous Hg from sorbent media is acceptable provided that the analysis can meet the performance criteria in Table 12B-1 in Section 9 of this performance specification. Candidate recovery techniques include leaching, digestion, and thermal desorption. Candidate analytical techniques include ultraviolet atomic fluorescence (UV AF); ultraviolet atomic absorption (UV AA), with and without gold trapping; and in-situ X-ray fluorescence (XRF).

## 7.0 Reagents and Standards

Only NIST-certified or NIST-traceable calibration gas standards and reagents must be used for the tests and procedures required under this performance specification. The sorbent media may be any collection material (e.g., carbon, chemically-treated filter, etc.) capable of quantitatively capturing and recovering for subsequent analysis, all gaseous forms of Hg in the emissions from the intended application. Selection of the sorbent media must be based on the material's ability to achieve the performance criteria contained in this method as well as the sorbent's vapor phase Hg capture efficiency for the emissions matrix and the expected sampling duration at the test site.

## 8.0 Performance Specification Test Procedure

### 8.1 Installation and Measurement Location Specifications.

8.1.1 Selection of Monitoring Site. Sampling site information should be obtained in accordance with Method 1 in appendix A-1 to this part. Place the probe inlet at a point or location in the stack (or vent) downstream of all pollution control equipment and representative of the stack gas concentration of Hg. A location that has been shown to be free of stratification for Hg or, alternatively, SO<sub>2</sub> is recommended. An estimation of the expected stack Hg concentration is required to establish a target sample flow rate, total gas sample volume, and the mass of Hg<sup>0</sup> to be spiked onto section 3 of each sorbent trap.

8.1.2 Pre-sampling Spiking of Sorbent Traps. Based on the estimated Hg concentration in the stack, the target sample rate and the target sampling duration, calculate the expected mass loading for section 1 of each sorbent trap (see Section 12.1 of this performance specification). The pre-sampling spike to be

added to section 3 of each sorbent trap must be within  $\pm 50$  percent of the expected section 1 mass loading. Spike section 3 of each sorbent trap at this level, as described in Section 6.1.3 of this performance specification. For each sorbent trap, keep a record of the mass of  $\text{Hg}^0$  added to section 3. This record must include, at a minimum, the identification number of the trap, the date and time of the spike, the name of the analyst performing the procedure, the method of spiking, the mass of  $\text{Hg}^0$  added to section 3 of the trap ( $\mu\text{g}$ ), and the supporting calculations.

8.1.3 Pre-monitoring Leak Check. Perform a leak check with the sorbent traps in place in the sampling system. Draw a vacuum in each sample train. Adjust the vacuum in each sample train to  $\sim 15$ " Hg. Use the gas flow meter to determine leak rate. The leakage rate must not exceed 4 percent of the target sampling rate. Once the leak check passes this criterion, carefully release the vacuum in the sample train, then seal the sorbent trap inlet until the probe is ready for insertion into the stack or duct.

8.1.4 Determination of Flue Gas Characteristics. Determine or measure the flue gas measurement environment characteristics (gas temperature, static pressure, gas velocity, stack moisture, etc.) in order to determine ancillary requirements such as probe heating requirements (if any), sampling rate, proportional sampling conditions, moisture management, etc.

## 8.2 Monitoring.

8.2.1 System Preparation and Initial Data Recording. Remove the plug from the end of each sorbent trap and store each plug in a clean sorbent trap storage container. Remove the stack or duct port cap and insert the probe(s) with the inlet(s) aligned perpendicular to the stack gas flow. Secure the probe(s) and ensure that no leakage occurs between the duct and environment. Record initial data including the sorbent trap ID, start time, starting gas flow meter readings, initial temperatures, set points, and any other appropriate information.

8.2.2 Flow Rate Control. Set the initial sample flow rate at the target value from section 8.1.1 of this performance specification. Then, for every operating hour during the sampling period, record the date and time, the sample flow rate, the gas flow meter reading, the stack temperature (if needed), the flow meter temperatures (if needed), temperatures of heated

equipment such as the vacuum lines and the probes (if heated), and the sampling system vacuum readings. Also, record the stack gas flow rate and the ratio of the stack gas flow rate to the sample flow rate. Adjust the sampling flow rate to maintain proportional sampling, i.e., keep the ratio of the stack gas flow rate to sample flow rate within  $\pm 25$  percent of the reference ratio from the first hour of the data collection period (see section 12.2 of this performance specification). The sample flow rate through a sorbent trap monitoring system during any hour (or portion of an hour) that the unit is not operating must be zero.

8.2.3 Stack Gas Moisture Determination. If data from the sorbent trap monitoring system will be used to calculate Hg mass emissions, determine the stack gas moisture content using a continuous moisture monitoring system or other means acceptable to the Administrator, such as the ones described in §75.11(b) of this chapter. Alternatively, for combustion of coal, wood, or natural gas in boilers only, a default moisture percentage from §75.11(b) of this chapter may be used.

8.2.4 Essential Operating Data. Obtain and record any essential operating data for the facility during the test period, e.g., the barometric pressure for correcting the sample volume measured by a dry gas meter to standard conditions. At the end of the data collection period, record the final gas flow meter reading and the final values of all other essential parameters.

8.2.5 Post-monitoring Leak Check. When the monitoring period is completed, turn off the sample pump, remove the probe/sorbent trap from the port and carefully re-plug the end of each sorbent trap. Perform a leak check with the sorbent traps in place, at the maximum vacuum reached during the monitoring period. Use the same general approach described in section 8.1.3 of this performance specification. Record the leakage rate and vacuum. The leakage rate must not exceed 4 percent of the average sampling rate for the monitoring period. Following the leak check, carefully release the vacuum in the sample train.

8.2.6 Sample Recovery. Recover each sampled sorbent trap by removing it from the probe and seal both ends. Wipe any deposited material from the outside of the sorbent trap. Place the sorbent trap into an appropriate sample storage container and store/preserve it in an appropriate manner.



8.2.7 Sample Preservation, Storage, and Transport. While the performance criteria of this approach provide for verification of appropriate sample handling, it is still important that the user consider, determine, and plan for suitable sample preservation, storage, transport, and holding times for these measurements. Therefore, procedures in recognized voluntary consensus standards such as those in ASTM D6911-03 "Standard Guide for Packaging and Shipping Environmental Samples for Laboratory Analysis" should be followed for all samples.

8.2.8 Sample Custody. Proper procedures and documentation for sample chain of custody are critical to ensuring data integrity. Chain of custody procedures in recognized voluntary consensus standards such as those in ASTM D4840-99 "Standard Guide for Sample Chain-of-Custody Procedures" should be followed for all samples (including field samples and blanks).

### 8.3 Relative Accuracy (RA) Test Procedure

8.3.1 For the initial certification of a sorbent trap monitoring system, a RA Test is required. Follow the basic RA test procedures and calculation methodology described in Sections 8.4.1 through 8.4.7 and 12.4 of PS 12A in this appendix, replacing the term "CEMS" with "sorbent trap monitoring system".

8.3.2 Special Considerations. The type of sorbent material used in the traps must be the same as that used for daily operation of the monitoring system; however, the size of the traps used for the RA test may be smaller than the traps used for daily operation of the system. Spike the third section of each sorbent trap with elemental Hg, as described in section 8.1.2 of this performance specification. Install a new pair of sorbent traps prior to each test run. For each run, the sorbent trap data must be validated according to the quality assurance criteria in Table 12B-1 in Section 9.0, below.

8.3.3 Acceptance Criteria. The RA of the sorbent trap monitoring system must be no greater than 20 percent of the mean value of the RM test data in terms of units of  $\mu\text{g}/\text{scm}$ . Alternatively, if the RM concentration is less than or equal to  $5.0 \mu\text{g}/\text{scm}$ , then the RA results are acceptable if the absolute difference between the means of the RM and sorbent trap monitoring system values does not exceed  $1.0 \mu\text{g}/\text{scm}$ .

## 9.0 Quality Assurance and Quality Control (QA/QC)

Table 12B-1 summarizes the QA/QC performance criteria that are used to validate the Hg emissions data from a sorbent trap monitoring system. Failure to achieve these performance criteria will result in invalidation of Hg emissions data, except where otherwise noted.

**TABLE 12B-1. QA/QC CRITERIA FOR SORBENT TRAP MONITORING SYSTEM OPERATION AND CERTIFICATION**

QA/QC Test or Specification	Acceptance Criteria	Frequency	Consequences if Not Met
Pre-monitoring leak check	≤4% of target sampling rate	Prior to monitoring	Monitoring must not commence until the leak check is passed
Post-monitoring leak check	≤4% of average sampling rate	After monitoring	Invalidate the data from the paired traps or, if certain conditions are met, report adjusted data from a single trap (see Section 12.7.1.3)
Ratio of stack gas flow rate to sample flow rate	Hourly ratio may not deviate from the reference ratio by more than ± 25%.	Every hour throughout monitoring period	Invalidate the data from the paired traps or, if certain conditions are met, report adjusted data from a single trap (see Section 12.7.1.3)
Sorbent trap section 2 breakthrough	≤5% of Section 1 Hg mass	Every sample	Invalidate the data from the paired traps or, if certain conditions are met, report adjusted data from a single trap (see Section 12.7.1.3)
Paired sorbent trap agreement	<p>≤10% Relative Deviation (RD) if the average concentration is &gt; 1.0 µg/m<sup>3</sup></p> <p>≤ 20% RD if the average concentration is ≤ 1.0 µg/m<sup>3</sup></p> <p>Results also acceptable if absolute difference between concentrations from paired traps is ≤ 0.03 µg/m<sup>3</sup></p>	Every sample	Either invalidate the data from the paired traps or report the results from the trap with the higher Hg concentration
Spike Recovery Study	Average recovery between 85% and 115% for each of the 3 spike concentration levels	Prior to analyzing field samples and prior to use of new sorbent media	Field samples must not be analyzed until the percent recovery criteria has been met
Multipoint analyzer calibration	Each analyzer reading within ± 10% of true value and	On the day of analysis, before	Recalibrate until successful

	$r^2 \geq 0.99$	analyzing any samples	
Analysis of independent calibration standard	Within $\pm 10\%$ of true value	Following daily calibration, prior to analyzing field samples	Recalibrate and repeat independent standard analysis until successful
Spike recovery from section 3 of both sorbent traps	75-125% of spike amount	Every sample	Invalidate the data from the paired traps or, if certain conditions are met, report adjusted data from a single trap (see Section 12.7.1.3)
Relative Accuracy	RA $\leq 20.0\%$ of RM mean value; or if RM mean value $\leq 5.0 \mu\text{g}/\text{scm}$ , absolute difference between RM and sorbent trap monitoring system mean values $\leq 1.0 \mu\text{g}/\text{scm}$	RA specification must be met for initial certification	Data from the system are invalid until a RA test is passed
Gas flow meter calibration	An initial calibration factor (Y) has been determined at 3 settings; for mass flow meters, initial calibration with stack gas has been performed. For subsequent calibrations, Y within $\pm 5\%$ of average value from the most recent 3-point calibration	At 3 settings prior to initial use and at least quarterly at one setting thereafter	Recalibrate meter at 3 settings to determine a new value of Y
Temperature sensor calibration	Absolute temperature measured by sensor within $\pm 1.5\%$ of a reference sensor	Prior to initial use and at least quarterly thereafter	Recalibrate; sensor may not be used until specification is met
Barometer calibration	Absolute pressure measured by instrument within $\pm 10 \text{ mm Hg}$ of reading with a NIST-traceable barometer	Prior to initial use and at least quarterly thereafter	Recalibrate; instrument may not be used until specification is met

## 10.0 Calibration and Standardization

10.1 Gaseous and Liquid Standards. Only NIST certified or NIST-traceable calibration standards (i.e., calibration gases, solutions, etc.) must be used for the spiking and analytical procedures in this performance specification.

10.2 Gas Flow Meter Calibration. The manufacturer or supplier of the gas flow meter should perform all necessary set-up, testing, programming, etc., and should provide the end user with any necessary instructions, to ensure that the meter will give an accurate readout of dry gas volume in standard cubic meters for the particular field application.

10.2.1.3 Initial Calibration Factor. Calculate an individual calibration factor  $Y_i$  at each tested flow rate from section 10.2.1.1 or 10.2.1.2 of this performance specification (as applicable), by taking the ratio of the reference sample volume to the sample volume recorded by the gas flow meter. Average the three  $Y_i$  values, to determine  $Y$ , the calibration factor for the flow meter. Each of the three individual values of  $Y_i$  must be within  $\pm 0.02$  of  $Y$ . Except as otherwise provided in sections 10.2.1.4 and 10.2.1.5 of this performance specification, use the average  $Y$  value from the three level calibration to adjust all subsequent gas volume measurements made with the gas flow meter.

10.2.2 Initial On-Site Calibration Check. For a mass flow meter that was initially calibrated using a compressed gas mixture, an on-site calibration check must be performed before using the flow meter to provide data. While sampling stack gas, check the calibration of the flow meter at one intermediate flow rate typical of normal operation of the monitoring system. Follow the basic procedures in section 10.2.1.1 or 10.2.1.2 of this performance specification. If the onsite calibration check shows that the value of  $Y_i$ , the calibration factor at the tested flow rate, differs by more than 5 percent from the value of  $Y$  obtained in the initial calibration of the meter, repeat the full 3-level calibration of the meter using stack gas to determine a new value of  $Y$ , and apply the new  $Y$  value to all subsequent gas volume measurements made with the gas flow meter.

10.2.3 Ongoing Quality Control. Recalibrate the gas flow meter quarterly at one intermediate flow rate setting representative of normal operation of the monitoring system. Follow the basic procedures in section 10.2.1.1 or 10.2.1.2 of this performance specification. If a quarterly recalibration shows that the value of  $Y_i$ , the calibration factor at the tested flow rate, differs from the current value of  $Y$  by more than 5 percent, repeat the full 3-level calibration of the meter to determine a new value of  $Y$ , and apply the new  $Y$  value to all subsequent gas volume measurements made with the gas flow meter.

10.3 Calibration of Thermocouples and Other Temperature Sensors. Use the procedures and criteria in section 10.3 of Method 2 in appendix A-1 to this part to calibrate in-stack temperature sensors and thermocouples. Calibrations must be performed prior to initial use and at least quarterly thereafter. At each calibration point, the absolute temperature measured by the temperature sensor must agree to within  $\pm 1.5$

percent of the temperature measured with the reference sensor, otherwise the sensor may not continue to be used.

10.4 Barometer Calibration. Calibrate the barometer against another barometer that has a NIST-traceable calibration. This calibration must be performed prior to initial use and at least quarterly thereafter. At each calibration point, the absolute pressure measured by the barometer must agree to within  $\pm 10$  mm Hg of the pressure measured by the NIST-traceable barometer, otherwise the barometer may not continue to be used.

10.5 Calibration of Other Sensors and Gauges. Calibrate all other sensors and gauges according to the procedures specified by the instrument manufacturer(s).

10.6 Analytical System Calibration. See section 11.1 of this performance specification.

## 11.0 Analytical Procedures

The analysis of the Hg samples may be conducted using any instrument or technology capable of quantifying total Hg from the sorbent media and meeting the performance criteria in section 9 of this performance specification.

11.1 Analyzer System Calibration. Perform a multipoint calibration of the analyzer at three or more upscale points over the desired quantitative range (multiple calibration ranges must be calibrated, if necessary). The field samples analyzed must fall within a calibrated, quantitative range and meet the necessary performance criteria. For samples that are suitable for aliquotting, a series of dilutions may be needed to ensure that the samples fall within a calibrated range. However, for sorbent media samples that are consumed during analysis (e.g., thermal desorption techniques), extra care must be taken to ensure that the analytical system is appropriately calibrated prior to sample analysis. The calibration curve range(s) should be determined based on the anticipated level of Hg mass on the sorbent media. Knowledge of estimated stack Hg concentrations and total sample volume may be required prior to analysis. The calibration curve for use with the various analytical techniques (e.g., UV AA, UV AF, and XRF) can be generated by directly introducing standard solutions into the analyzer or by spiking the standards onto the sorbent media and then introducing into the analyzer after preparing the sorbent/standard according to the particular analytical technique. For each calibration curve, the value of the square of the linear correlation coefficient,

i.e.,  $r^2$ , must be  $\geq 0.99$ , and the analyzer response must be within  $\pm 10$  percent of reference value at each upscale calibration point. Calibrations must be performed on the day of the analysis, before analyzing any of the samples. Following calibration, an independently prepared standard (not from same calibration stock solution) must be analyzed. The measured value of the independently prepared standard must be within  $\pm 10$  percent of the expected value.

11.2 Sample Preparation. Carefully separate the three sections of each sorbent trap. Combine for analysis all materials associated with each section, i.e., any supporting substrate that the sample gas passes through prior to entering a media section (e.g., glass wool, polyurethane foam, etc.) must be analyzed with that segment.

11.3 Spike Recovery Study. Before analyzing any field samples, the laboratory must demonstrate the ability to recover and quantify Hg from the sorbent media by performing the following spike recovery study for sorbent media traps spiked with elemental mercury. Using the procedures described in sections 6.2 and 12.1 of this performance specification, spike the third section of nine sorbent traps with gaseous  $\text{Hg}^0$ , i.e., three traps at each of three different mass loadings, representing the range of masses anticipated in the field samples. This will yield a 3 x 3 sample matrix. Prepare and analyze the third section of each spiked trap, using the techniques that will be used to prepare and analyze the field samples. The average recovery for each spike concentration must be between 85 and 115 percent. If multiple types of sorbent media are to be analyzed, a separate spike recovery study is required for each sorbent material. If multiple ranges are calibrated, a separate spike recovery study is required for each range.

11.4 Field Sample Analyses. Analyze the sorbent trap samples following the same procedures that were used for conducting the spike recovery study. The three sections of each sorbent trap must be analyzed separately (i.e., section 1, then section 2, then section 3). Quantify the total mass of Hg for each section based on analytical system response and the calibration curve from section 11.1 of this performance specification. Determine the spike recovery from sorbent trap section 3. The spike recovery must be no less than 75 percent and no greater than 125 percent. To report the final Hg mass for each trap, add together the Hg masses collected in trap sections 1 and 2.

## 12.0 Calculations, Data Reduction, and Data Analysis

12.1 Calculation of Pre-Sampling Spiking Level. Determine sorbent trap section 3 spiking level using estimates of the stack Hg concentration, the target sample flow rate, and the expected monitoring period. Calculate  $M_{exp}$ , the expected Hg mass that will be collected in section 1 of the trap, using Equation 12B-1. The pre-sampling spike must be within  $\pm 50$  percent of this mass.

$$M_{exp} = [Q_s t_s C_{est}] \times 10^{-3} \quad (\text{Equation 12B-1})$$

Where:

$M_{exp}$  = Expected sample mass ( $\mu\text{g}$ )

$Q_s$  = Sample flow rate (L/min)

$t_s$  = Expected monitoring period (min)

$C_{est}$  = Estimated Hg concentration in stack gas ( $\mu\text{g}/\text{m}^3$ )

$10^{-3}$  = Conversion factor ( $\text{m}^3/\text{L}$ )

Example calculation: For an estimated stack Hg concentration of  $5 \mu\text{g}/\text{m}^3$ , a target sample rate of 0.30 L/min; and a monitoring period of 5 days:

$$M_{exp} = (0.30 \text{ L/min})(1440 \text{ min/day})(5 \text{ days})(10^{-3} \text{ m}^3/\text{L})(5 \mu\text{g}/\text{m}^3) = 10.8 \mu\text{g}$$

A pre-sampling spike of  $10.8 \mu\text{g} \pm 50$  percent is, therefore, appropriate.

12.2 Calculations for Flow-Proportional Sampling. For the first hour of the data collection period, determine the reference ratio of the stack gas volumetric flow rate to the sample flow rate, as follows:

$$R_{ref} = \frac{KQ_{ref}}{F_{ref}} \quad (\text{Equation 12B-2})$$

Where:

$R_{ref}$  = Reference ratio of hourly stack gas flow rate to hourly sample flow rate

$Q_{ref}$  = Average stack gas volumetric flow rate for first hour of collection period (scfh)

$F_{ref}$  = Average sample flow rate for first hour of the collection period, in appropriate units (e.g.,

liters/min, cc/min, dscm/min)

K = Power of ten multiplier, to keep the value of  $R_{ref}$  between 1 and 100. The appropriate K value will depend on the selected units of measure for the sample flow rate.

Then, for each subsequent hour of the data collection period, calculate ratio of the stack gas flow rate to the sample flow rate using Equation 12B-3:

$$R_h = \frac{KQ_h}{F_h} \quad (\text{Equation 12B-3})$$

Where:

$R_h$  = Ratio of hourly stack gas flow rate to hourly sample flow rate

$Q_h$  = Average stack gas volumetric flow rate for the hour (scfh)

$F_h$  = Average sample flow rate for the hour, in appropriate units (e.g., liters/min, cc/min, dscm/min)

K = Power of ten multiplier, to keep the value of  $R_h$  between 1 and 100. The appropriate K value will depend on the selected units of measure for the sample flow rate and the range of expected stack gas flow rates.

Maintain the value of  $R_h$  within  $\pm 25$  percent of  $R_{ref}$  throughout the data collection period.

12.3 Calculation of Spike Recovery. Calculate the percent recovery of each section 3 spike, as follows:

$$\%R = \frac{M_3}{M_s} \times 100 \quad (\text{Equation 12B-4})$$

Where:

$\%R$  = Percentage recovery of the pre-sampling spike

$M_3$  = Mass of Hg recovered from section 3 of the sorbent trap, ( $\mu\text{g}$ )

$M_s$  = Calculated Hg mass of the pre-sampling spike, from section 8.1.2 of this performance specification, ( $\mu\text{g}$ )

12.4 Calculation of Breakthrough. Calculate the percent breakthrough to the second section of the sorbent trap, as follows:

$$\%B = \frac{M_2}{M_1} \times 100 \quad (\text{Equation 12B-5})$$



Where:

%B = Percent breakthrough

M<sub>2</sub> = Mass of Hg recovered from section 2 of the sorbent trap, (µg)

M<sub>1</sub> = Mass of Hg recovered from section 1 of the sorbent trap, (µg)

12.5 Calculation of Hg Concentration. Calculate the Hg concentration for each sorbent trap, using the following equation:

$$C = \frac{M^*}{V_t} \quad (\text{Equation 12B-6})$$

Where:

C = Concentration of Hg for the collection period, (µg/dscm)

M\* = Total mass of Hg recovered from sections 1 and 2 of the sorbent trap, (µg)

V<sub>t</sub> = Total volume of dry gas metered during the collection period, (dscm). For the purposes of this performance specification, standard temperature and pressure are defined as 20 °C and 760 mm Hg, respectively.

12.6 Calculation of Paired Trap Agreement. Calculate the relative deviation (RD) between the Hg concentrations measured with the paired sorbent traps:

$$RD = \frac{|C_a - C_b|}{C_a + C_b} \times 100 \quad (\text{Equation 12B-7})$$

Where:

RD = Relative deviation between the Hg concentrations from traps "a" and "b" (percent)

C<sub>a</sub> = Concentration of Hg for the collection period, for sorbent trap "a" (µg/dscm)

C<sub>b</sub> = Concentration of Hg for the collection period, for sorbent trap "b" (µg/dscm)

12.7 Calculation of Relative Accuracy. Calculate the relative accuracy as described in Section 12.4 of PS 12A in this appendix.

12.8 Data Reduction. Typical monitoring periods for normal, day-to-day operation of a sorbent trap monitoring system range from about 24 hours to 168 hours. For the required RA tests of the system, smaller sorbent traps are often used, and the "monitoring period" or time per run is considerably shorter (e.g., 1 hour or less). Generally speaking, to validate sorbent trap monitoring system data, the acceptance criteria for the following five QC specifications in Table 12B-1 above must be met for both traps: (a) the post-monitoring leak check; (b) the ratio of stack gas flow rate to sample flow rate; (c) section 2 breakthrough; (d) paired trap agreement; and (e) section 3 spike recovery.

12.8.1 For routine day-to-day operation of a sorbent trap monitoring system, when both traps meet the acceptance criteria for all five QC specifications, the two measured Hg concentrations must be averaged arithmetically and the average value must be applied to each hour of the data collection period.

12.8.2 To validate a RA test run, both traps must meet the acceptance criteria for all five QC specifications. However, as specified in Section 12.8.3 below, for routine day-to-day operation of the monitoring system, a monitoring period may, in certain instances, be validated based on the results from one trap.

12.8.3 For the routine, day-to-day operation of the monitoring system, when one of the two sorbent trap samples or sampling systems either: (a) fails the post-monitoring leak check; or (b) has excessive section 2 breakthrough; or (c) fails to maintain the proper stack flow-to-sample flow ratio; or (d) fails to achieve the required section 3 spike recovery, provided that the other trap meets the acceptance criteria for all four of these QC specifications, the Hg concentration measured by the valid trap may multiplied by a factor of 1.111 and then used for reporting purposes. Further, if both traps meet the acceptance criteria for all four of these QC specifications, but the acceptance criterion for paired trap agreement is not met, the owner or operator may report the higher of the two Hg concentrations measured by the traps, in lieu of invalidating the data from the paired traps.

12.8.4 Whenever the data from a pair of sorbent traps must be invalidated and no quality-assured data from a certified backup Hg monitoring system or Hg reference method are available to cover the hours in the data collection period, treat those hours in the manner specified in the applicable regulation (i.e., use missing data substitution procedures or count the hours as monitoring system down time, as appropriate).

### *13.0 Monitoring System Performance*

These monitoring criteria and procedures have been successfully applied to coal-fired utility boilers (including units with post-combustion emission controls), having vapor-phase Hg concentrations ranging from 0.03  $\mu\text{g/dscm}$  to approximately 100  $\mu\text{g/dscm}$ .

### *14.0 Pollution Prevention [Reserved]*

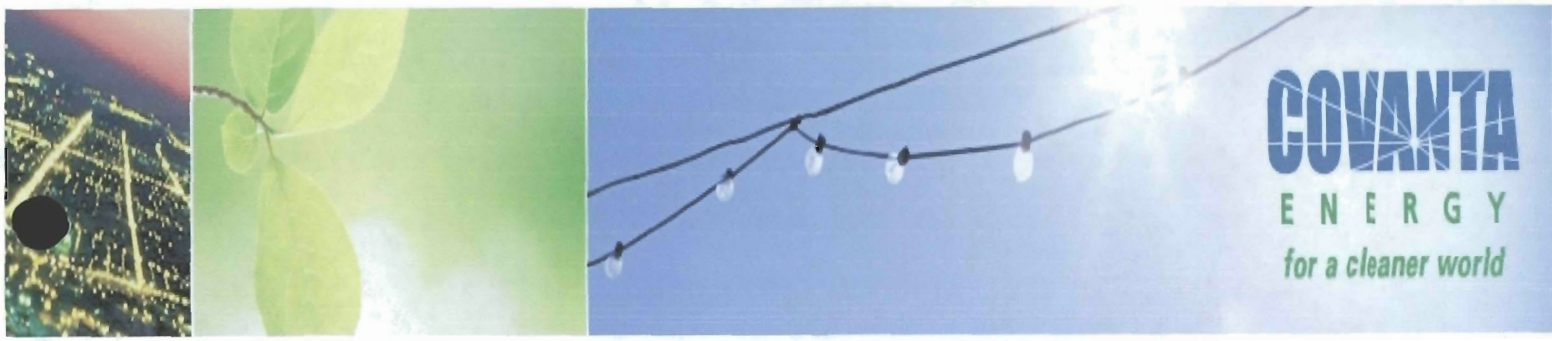
### *15.0 Waste Management [Reserved]*

### *16.0 Alternative Procedures [Reserved]*

### *17.0 Bibliography*

17.1 40 CFR Part 60, Appendix B, "Performance Specification 2 - Specifications and Test Procedures for SO<sub>2</sub> and NO<sub>x</sub> Continuous Emission Monitoring Systems in Stationary Sources."

17.2 40 CFR Part 60, Appendix B, "Performance Specification 12A - Specifications and Test Procedures for Total Vapor Phase Mercury Continuous Emission Monitoring Systems in Stationary Sources."



## ATTACHMENT 3

- ETV Certificate and Reports for HG-324K Mercury Monitor



## Mercury Continuous Emission Monitors (CEMs)

The U.S. EPA Environmental Technology Verification (ETV) Program's Advanced Monitoring Systems (AMS) Center, operated by Battelle under a cooperative agreement with EPA, has verified the performance of seven continuous emission monitors<sup>1</sup> (CEMs) for measuring mercury emissions (**Figure 1**). Four additional monitoring technologies are currently in testing with reports to be final in early 2007. To address the health effects caused by mercury emissions from coal-fired plants, EPA recently issued the Clean Air Mercury Rule (CAMR). This rule requires coal-fired power plants, the largest remaining unregulated source of human-generated mercury emissions in the U.S., to reduce mercury emissions. The rule also will require power plants to monitor their mercury emissions using technologies like those verified by the ETV Program.



One of the test locations for mercury CEM verification

### Technology Description and Verification Testing

CEMs for mercury are a relatively new technology category. They offer an advantage over conventional laboratory techniques (e.g., the Ontario Hydro method) in that they can provide continuous or frequent results through sequential readings at intervals of several minutes,

and thus, they avoid the delay, labor, and cost associated with laboratory methods.

The ETV-verified CEMs determine elemental mercury vapor concentrations by atomic absorption (AA), atomic fluorescence (AF), or plasma atomic emission (AE). The CEMs use aqueous reagents or heated catalysts to reduce oxidized forms of mercury to elemental mercury for detection, allowing measurement of total vapor-phase mercury. Although some CEMs only measure total vapor-phase mercury (i.e., the sum of elemental and oxidized mercury vapor), others allow separate measurement of the elemental and oxidized forms. **Table 1** summarizes some of the performance data for the verified technologies. Additional information on the verification of mercury CEMs can be found at <http://www.epa.gov/etv/vt-ams.html> under the mercury emissions monitors category.

The verification testing was conducted in two phases. In the first phase, four of the technologies were tested under conditions simulating a) coal-fired flue gas, and b) municipal incinerator flue gas. The tests took place at

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a pilot-scale incinerator in Research Triangle Park, North Carolina, over a three-week period. In the second phase, five technologies (including two of the technologies tested in the first phase) were evaluated at a full-scale hazardous waste incinerator in Oak Ridge, Tennessee. In addition, the ETV Program is currently conducting a third phase of testing at a coal-fired power plant. The box on the right identifies CEMs and sorbent-based sampling technologies included in this third phase.

### Mercury and Its Regulatory Background at a Glance

Mercury is a toxic, persistent pollutant that, after deposition from the atmosphere and methylation bioaccumulates in the food chain, particularly in fish. Mercury can cause adverse neurological health effects, particularly in young children and the unborn children of mothers who eat food with significant quantities of mercury.

The Clean Air Mercury Rule (CAMR), which EPA issued on March 18, 2005, creates a market-based cap-and-trade program that will reduce nationwide utility emissions of mercury. Under a cap-and-trade program, coal-fired power plants that reduce emissions more than is required receive allowances. They can then trade these allowances to sources that are unable to meet the requirement, or bank them for future use.

A cap-and-trade program, like that under the CAMR, must include reliable monitoring of emissions to ensure that reductions occur, allow for tracking progress, and lend credibility to the trading component of the program. Therefore, the CAMR requires coal-fired utilities that emit more than 29 pounds of mercury per year to collect mercury emission data continuously. To collect these data, the utilities can use either CEMs, like those verified by the ETV Program, or another long-term mercury sampling method, a sorbent trap monitoring approach.

#### Mercury Monitoring Technologies Included in the Third Phase of ETV Verification

**Tekran Instruments**, Series 3300 Mercury CEM

**Thermo Electron**, Mercury Freedom System  
**Environmental Supply Company**, HG-324 sorbent-based sampling system

**Apex Instruments**, mercury sorbent-based sampling system

<sup>1</sup> The ETV Program operates largely as a public-private partnership through competitive cooperative agreements with non-profit research institutes. The program provides objective quality-assured data on the performance of commercial-ready technologies. Verification does not imply product approval or effectiveness. ETV does not endorse the purchase or sale of any products and services mentioned in this document.

### Selected Outcomes of Verified Mercury CEM Technologies

- Contributed to advancing mercury monitoring technology and resulted in improvements in monitors by the participating vendors
- Helped inform the development of the CAMR and could assist in future rule refinements
- Helped small vendors compete in the marketplace
- Verification of the mercury CEMs involved significant collaboration with state agencies (e.g., Massachusetts and Connecticut), the Department of Energy and Illinois Clean Coal Institute. These collaborations resulted in the sharing of scientific expertise among the agencies and enabled smaller vendors to participate in the tests.

Fig 1. ETV-Verified CEMs for Mercury (First Two Phases of Testing)

**Envimetrics, Argus-Hg 1000 Mercury CEM:** Uses AE spectroscopy with a proprietary catalytic converter that reduces molecular forms of mercury to atomic mercury. Total mercury can be measured during automatic operation, or both total and elemental mercury can be measured when manually operated.

**OPSIS AB, HG-200 Mercury CEM:** Uses a double-beam photometer to measure total or elemental mercury with a thermocatalytic converter that forms elemental mercury from any oxidized mercury compounds to measure total mercury.

**Nippon Instruments Corporation, DM-6/DM-6P Mercury CEM:** Uses cold vapor AA with a catalytic process to measure total mercury.

**PS Analytical, Ltd., Sir Galahad II Mercury CEM (verified in both phases):** Uses AF to provide separate and continuous measurement of elemental and total mercury with a proprietary aqueous reagent to convert oxidized mercury to elemental mercury for total mercury measurement.

**Nippon Instruments Corporation, AM-2 Elemental Mercury CEM:** Uses cold vapor AA, with a distilled water scrubbing trap for removal of any oxidized mercury species, to measure elemental mercury.

**CEM:** Uses cold vapor AA to provide separate and continuous measurements of elemental and total mercury, with catalytic pyrolysis to decompose oxidized mercury to elemental mercury for total mercury measurement.

**Nippon Instruments Corporation, MS-1/DM-5 Mercury CEM (verified in both phases):** Uses cold vapor AA to provide separate and continuous measurements of elemental and oxidized mercury, which are separated using a wet scrubbing and chemical reaction system.

The price of the monitors ranged from \$30,000 to \$70,000 at the time of testing.

Table 1. Selected Performance of Verified CEMs for Mercury

Technology <sup>A</sup>	Average Relative Accuracy, %	Relative Precision, %	Response Time (95%)	Bias, %	Correlation <sup>B</sup>			Data Completeness
					Slope	Intercept	r <sup>2</sup>	
First Phase								
A	58.2 to 71% (total mercury)	2.5 to 27%	30 to 100 seconds	-44.5 to -20.5%	not reported	not reported	0.621	Not estimated
B	14 to 23% (elemental mercury)	3 to 40.3%	One 13-minute cycle	7%	0.885	-0.212	0.973	100%
C (Phase I)	20.6 to 32.8% (total mercury)	1.8 to 24.7%	One 5- to 6-minute cycle	-4.9 to -0.3%	0.681	2.492	0.978	100%
D (Phase I)	13.2 to 39.1% (total mercury)	3.7 to 23.9%	35 to 50 seconds	-7%	0.607	3.92	0.938	100%
Second Phase								
C (Phase II)	59.8% (total mercury)	8.9 to 15.9%	One 5- to 6-minute cycle	2.8 to 6.9%	0.4973	6.8904	0.875	88.3%
D (Phase II)	11.2% (total mercury)	9.2 to 17.3%	2 to 3 minutes	0.0 to 6.6%	0.899	2.4969	0.987	97.7%
E	76.5% (overall)	10.1 to 22.1%	One 7-minute cycle	0.3 to 14.6%	0.3404	9.4121	0.839	92.7%
F	20.3% (overall)	9.1 to 10.9%	2 minutes	0.0 to 13.6%	0.8347	3.5033	0.953	97.5%
G	76.3% (overall)	12.5 to 43.3%	One 5-minute cycle	Not evaluated	0.3559	8.1695	0.935	65.8%

<sup>A</sup> Because the ETV Program does not compare technologies, the performance results shown in this table do not identify the vendor associated with each result and are not in the same order as the list of technologies in Figure 1.

<sup>B</sup> Correlation data shown are for total mercury, except technology B, where results shown are for elemental mercury.

Note: In each phase of verification testing, the Ontario Hydro method was used as the reference method for establishing the performance of the tested technologies. The performance parameters verified included the following: accuracy relative to the Ontario Hydro method, correlation with that method, precision (i.e., repeatability), bias, calibration/zero drift, response time, interferences, data completeness, and other operational factors. The ETV Program found that the average relative accuracy for the monitors ranged from 11.2 to 76.5%. A result of 0% indicates perfect accuracy relative to the reference mercury concentration. The relative precision ranged from 1.8 to 43.3%. A result of 0% indicates perfect precision. A higher r<sup>2</sup> value indicates a higher correlation with the standard test method over the range of concentrations tested.

### References

- U.S. EPA, Mercury, <http://www.epa.gov/mercury/>.
- U.S. EPA, 2006. *ETV Case Studies: Demonstrating Program Outcomes, Volume II*. EPA/600/R-06/082. September 2006. (Primary source)
- U.S. EPA, ETV, <http://www.epa.gov/etv/>.

## THE ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM



### ETV Joint Verification Statement

**TECHNOLOGY TYPE:** Mercury Emissions Monitor

**APPLICATION:** Sorbent Trap Monitoring of Flue Gas Mercury

**TECHNOLOGY NAME:** HG-324K System

**COMPANY:** Environmental Supply Company

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The U.S. Environmental Protection Agency (EPA) has established the Environmental Technology Verification (ETV) Program to facilitate the deployment of innovative or improved environmental technologies through performance verification and dissemination of information. The goal of the ETV Program is to further environmental protection by accelerating the acceptance and use of improved and cost-effective technologies. ETV seeks to achieve this goal by providing high-quality, peer-reviewed data on technology performance to those involved in the design, distribution, financing, permitting, purchase, and use of environmental technologies. Information and ETV documents are available at [www.epa.gov/etv](http://www.epa.gov/etv).

ETV works in partnership with recognized standards and testing organizations, with stakeholder groups (consisting of buyers, vendor organizations, and permittees), and with individual technology developers. The program evaluates the performance of innovative technologies by developing test plans that are responsive to the needs of stakeholders, conducting field or laboratory tests (as appropriate), collecting and analyzing data, and preparing peer-reviewed reports. All evaluations are conducted in accordance with rigorous quality assurance (QA) protocols to ensure that data of known and adequate quality are generated and that the results are defensible.

The Advanced Monitoring Systems (AMS) Center, one of six technology areas under ETV, is operated by Battelle in cooperation with EPA's National Exposure Research Laboratory. In collaboration with the Illinois Clean Coal Institute, and with assistance from the Northern Indiana Public Service Company, the AMS Center evaluated the performance of the Environmental Supply Company's HG-324K sorbent-based mercury sampling system for determining mercury in stack gas at a coal-fired power plant. This verification statement provides a summary of the test results.

## VERIFICATION TEST DESCRIPTION

The performance of the HG-324K was evaluated in terms of relative accuracy (RA), data completeness, and operational factors (ease of use, maintenance and data output needs, power and other consumables use, reliability, and operational costs). RA was determined according to Equation A-10 of Chapter 40 of the Code of Federal Regulations Part 75 (40 CFR Part 75) Appendix A, by comparing HG-324K vapor-phase total mercury ( $Hg_T$ ) results to simultaneous results from American Society for Testing and Materials (ASTM) D 6784-02, the "Ontario Hydro" (OH) method. Data completeness was assessed as the percentage of maximum data return achieved by the HG-324K over its test period. Operational factors were evaluated by means of observations during use and records of needed maintenance, vendor activities, and expendables use.

The HG-324K was verified at Unit 17 of the R.M. Schahfer Generating Station, located near Wheatfield, Indiana, between June 12 and 15, 2006. Unit 17 burns pulverized Illinois sub-bituminous coal and has an electrostatic precipitator and a wet flue gas desulfurization unit. During this period, twelve successive OH method runs, each of 2 hours duration, were conducted on the Unit 17 stack using paired OH trains. Those reference samples were collected and analyzed to determine elemental mercury and oxidized mercury, the sum of which is  $Hg_T$ .

QA oversight of verification testing was provided by Battelle and EPA. Battelle QA staff conducted a technical systems audit, a performance evaluation audit, and a data quality audit of 10% of the test data.

This verification statement, the full report on which it is based, and the test/QA plan for this verification test are all available at [www.epa.gov/etv/centers/center1.html](http://www.epa.gov/etv/centers/center1.html).

## TECHNOLOGY DESCRIPTION

The following description of the HG-324K is based on information provided by the vendor. The information provided below was not verified in this test.

The HG-324K system was designed to sample mercury emissions from coal-fired sources as specified in Appendix K in 40 CFR Part 75. The system consists of a dual heated probe, knockout and drying impingers to remove moisture, a connecting umbilical, and the HG-324K automated sampler. An integrated sample of vapor phase mercury is captured on two parallel and independent sorbent traps that are placed in the stack on the front of the sampling probe. Stack gas is drawn through each of the traps at a constant flow rate of approximately 500 cubic centimeters per minute. The traps and probe are heated to prevent condensation of moisture from the sample gas. After exiting the probe, the sample gas passes through the knockout and drying impingers to remove moisture and then is drawn into the HG-324K sampler for measurement of the sample volume. The HG-324K provides proportional, integral, derivative flow control of the dual samples; records all temperatures including the stack, probe, and condenser; controls the probe temperature; and measures the dry standard volume of sample gas.

The HG-324K is controlled using an industrial data acquisition and control system with a removable CompactFlash™ memory card for storing data files. The HG-324K may be connected to a plant network through wireless or direct connection to allow program control and remote data access. It comes in a watertight, corrosion proof case with 2-inch hard rubber transport wheels and a retractable extension handle. The outside dimensions are 24-5/8 inches long by 19-1/2 inches wide by 14 inches deep.

The sorbent traps used with the HG-324K were prepared and analyzed for  $Hg_T$  by Frontier Geosciences. Consistent with Appendix K, each trap incorporated a breakthrough section and a pre-spiked section to assess mercury spike recovery.



## VERIFICATION RESULTS

The RA of the HG-324K for determining  $Hg_T$  was 29.5%, based on 11 OH runs, when the comparison was based on HG-324K results corrected for trap blanks but not corrected for mercury spike recovery. For those 11 runs, the overall average  $Hg_T$  value from the OH reference method was 0.821 microgram per dry standard cubic meter ( $\mu\text{g}/\text{dscm}$ ), whereas that from the HG-324K was 1.004  $\mu\text{g}/\text{dscm}$ , a difference of 0.183  $\mu\text{g}/\text{dscm}$ . When comparing HG-324K results corrected for mercury spike recovery, the RA for 11 OH runs was 37.0%, and the OH and HG-324K average values were 0.821  $\mu\text{g}/\text{dscm}$  and 1.064  $\mu\text{g}/\text{dscm}$ , respectively, a difference of 0.243  $\mu\text{g}/\text{dscm}$ .

The HG-324K sampled during all 12 OH runs conducted over four days with no delays, breakdowns, broken traps, or sampling interruptions. The only problem encountered was that after Run 8 the post-test leak check failed. As a result, only 11 of the 12 sampling runs (91.7% data completeness) were suitable for comparison to the OH reference results.

The HG-324K was installed quickly and was operated by a vendor representative without serious problems. A failed post-test leak check in one sampling run was the only difficulty encountered. The sorbent traps were rugged and uniform in construction, so that no breakage occurred; no problems were encountered in placing the traps into the end of the sampling probe or recovering them after sampling. The sampling probe used with the HG-324K was simple and relatively light in weight, and was handled by a single operator in all sampling. The HG-324K sorbent sampling system incorporated data acquisition and transfer capabilities, including magnetic card recording media and wireless communication.

The cost of the HG-324K system as tested is \$18,750. As used in this test, the cost per sorbent trap sample was about \$500, including preparation of the trap, pre-spiking the trap with mercury, and analyzing the four-section trap after sampling.

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February 2007

# Environmental Technology Verification Report

ENVIRONMENTAL SUPPLY COMPANY  
HG-324K SORBENT-BASED MERCURY SAMPLING  
SYSTEM

Prepared by  
Battelle

**Battelle**  
*The Business of Innovation*

Under a cooperative agreement with

 **EPA** U.S. Environmental Protection Agency

ETV ✓ ETV ✓ ETV ✓

# **Environmental Technology Verification Report**

ETV Advanced Monitoring Systems Center

## **ENVIRONMENTAL SUPPLY COMPANY HG-324K SORBENT-BASED MERCURY SAMPLING SYSTEM**

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## Notice

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This report was prepared by Battelle to summarize testing supported in part by the Illinois Department of Commerce and Economic Opportunity through the Office of Coal Development and the Illinois Clean Coal Institute (ICCI). Neither Battelle nor any of its subcontractors nor the Illinois Department of Commerce and Economic Opportunity, Office of Coal Development; the ICCI; nor any person acting on behalf of either

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## Foreword

The U.S. Environmental Protection Agency (EPA) is charged by Congress with protecting the nation's air, water, and land resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. To meet this mandate, the EPA's Office of Research and Development provides data and science support that can be used to solve environmental problems and to build the scientific knowledge base needed to manage our ecological resources wisely, to understand how pollutants affect our health, and to prevent or reduce environmental risks.

The Environmental Technology Verification (ETV) Program has been established by the EPA to verify the performance characteristics of innovative environmental technology across all media and to report this objective information to permittees, buyers, and users of the technology, thus substantially accelerating the entrance of new environmental technologies into the marketplace. Verification organizations oversee and report verification activities based on testing and quality assurance protocols developed with input from major stakeholders and customer groups associated with the technology area. ETV consists of six environmental technology centers. Information about each of these centers can be found on the Internet at <http://www.epa.gov/etv/>.

Effective verifications of monitoring technologies are needed to assess environmental quality and to supply cost and performance data to select the most appropriate technology for that assessment. Under a cooperative agreement, Battelle has received EPA funding to plan, coordinate, and conduct such verification tests for "Advanced Monitoring Systems for Air, Water, and Soil" and report the results to the community at large. Information concerning this specific environmental technology area can be found on the Internet at <http://www.epa.gov/etv/centers/center1.html>.

## **Acknowledgments**

The authors wish to acknowledge the support of all those who helped plan and conduct the verification test, analyze the data, and prepare this report. This verification was funded in part by the Illinois Clean Coal Institute (ICCI) under Project No. 04-1/3.2D-1; we appreciate the involvement and support of Dr. Francois Botha, Project Manager for the ICCI. We acknowledge the contribution of the Northern Indiana Public Service Company (NIPSCo, a NiSource company) in hosting this verification at the R. M. Schahfer Generating Station and, in particular, the efforts of Craig Myers, Bert Valenkamph, and Gary Logan of NIPSCo in support of the field testing. We also thank Eric Ginsburg and William Grimley of U.S. EPA for their assistance in setting up a Site Access Agreement among EPA, Battelle, and NiSource. Finally, we would like to thank Robin Segall of U.S. EPA, Ernest Bouffard of the Connecticut Department of Environmental Protection, Francois Botha of ICCI, and Craig Myers of NIPSCo for their review of this verification report.

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## List of Abbreviations

agl	above ground level
AMS	Advanced Monitoring Systems
ASTM	American Society for Testing and Materials
CEM	continuous emission monitor
EPA	U.S. Environmental Protection Agency
ETV	Environmental Technology Verification
°F	degrees Fahrenheit
FGD	flue gas desulfurization
H <sub>2</sub> O <sub>2</sub>	hydrogen peroxide
H <sub>2</sub> SO <sub>4</sub>	sulfuric acid
Hg	mercury
HgCl <sub>2</sub>	mercuric chloride
Hg <sup>0</sup>	elemental mercury
Hg <sub>OX</sub>	oxidized mercury
Hg <sub>T</sub>	total mercury
HNO <sub>3</sub>	nitric acid
ICCI	Illinois Clean Coal Institute
KCl	potassium chloride
klb/hr	thousands of pounds per hour
KMnO <sub>4</sub>	potassium permanganate
L/min	liters per minute
MW	megawatt
µg/dscm	microgram per dry standard cubic meter
µg/mL	microgram per milliliter
mL	milliliter
NIST	National Institute of Standards and Technology
NO <sub>x</sub>	nitrogen oxides
OH	Ontario Hydro
ppm	part per million
PE	performance evaluation
QA	quality assurance
QC	quality control
QMP	quality management plan
RA	relative accuracy
RD	relative deviation

SO<sub>2</sub>  
TSA

sulfur dioxide  
technical systems audit

## **Chapter 1 Background**

The U.S. Environmental Protection Agency (EPA) supports the Environmental Technology Verification (ETV) Program to facilitate the deployment of innovative environmental technologies through performance verification and dissemination of information. The goal of the ETV Program is to further environmental protection by accelerating the acceptance and use of improved and cost-effective technologies. ETV seeks to achieve this goal by providing high-quality, peer-reviewed data on technology performance to those involved in the design, distribution, financing, permitting, purchase, and use of environmental technologies.

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The EPA's National Exposure Research Laboratory and its verification organization partner, Battelle, operate the Advanced Monitoring Systems (AMS) Center under ETV. The AMS Center recently evaluated the performance of the Environmental Supply Company's HG-324K sorbent-based mercury sampling system for determining mercury in stack gas at a coal-fired power plant. This evaluation was carried out in collaboration with the Illinois Clean Coal Institute and with the assistance of the Northern Indiana Public Service Company.

## Chapter 2 Technology Description

The objective of the ETV AMS Center is to verify the performance characteristics of environmental monitoring technologies for air, water, and soil. This report provides results for the verification testing of the Environmental Supply Company's HG-324K mercury sampling system. The following is a description of the HG-324K, based on information provided by the vendor. The information provided below was not verified in this test.

The HG-324K system (Figure 2-1) was designed to sample mercury emissions from coal-fired sources as specified in Appendix K in Chapter 40 of the Code of Federal Regulations Part 75 (40 CFR Part 75).<sup>(1)</sup> The system consists of a dual heated probe, knockout and drying impingers to remove moisture, a connecting umbilical, and the HG-324K automated sampler. An integrated sample of vapor phase mercury is captured on two parallel and independent sorbent traps that are placed in the stack on the front of the sampling probe. Stack gas is drawn through each of the traps at a constant flow rate of approximately 500 cubic centimeters per minute. The traps and probe are heated to prevent condensation of moisture from the sample gas. After exiting the probe, the sample gas passes through the knockout and drying impingers to remove moisture and

then is drawn into the HG-324K sampler for measurement of the sample volume. The HG-324K provides proportional, integral, derivative flow control of the dual samples; records all temperatures including the stack, probe, and condenser; controls the probe temperature; and measures the dry standard volume of sample gas.

The mass of mercury is determined using cold vapor atomic fluorescence spectrometry as specified in EPA Method 1631.<sup>(2)</sup> For quality control, each trap has a breakthrough section and a spike and recovery section. The concentration of vapor phase mercury in the stack is determined based on the mass of mercury captured on the sorbent trap and the dry standard stack gas volume measured by the HG-324K.

The HG-324K is controlled using an industrial data acquisition and control system with a removable CompactFlash™ memory card for storing data files. The HG-324K may be



**Figure 2-1. HG-324K Sorbent Tube Mercury Sampling System**

connected to a plant network through wireless or direct connection to allow program control and remote data access. It comes in a watertight, corrosion proof case with 2-inch hard rubber transport wheels and a retractable extension handle. The outside dimensions are 24-5/8 inches long by 19-1/2 inches wide by 14 inches deep.

The list price for the automated sampler is \$18,750. The sorbent traps used with the HG-324K in this test were prepared and analyzed by Frontier Geosciences, of Seattle, Washington. As used in this test, the cost per sorbent trap sample was about \$500, including preparation of the trap, pre-spiking with mercury, and analyzing the trap for mercury after sampling.

## Chapter 3

### Test Design and Procedures

#### 3.1 Introduction

This verification test was conducted according to procedures specified in the *Test/QA Plan for Verification of Continuous Emission Monitors (CEMs) and Sorbent-Based Samplers for Mercury at a Coal-Fired Power Plant*.<sup>(3)</sup> Appendix K of 40 CFR Part 75<sup>(1)</sup> establishes sorbent-based sampling systems as an acceptable approach for determining mercury in the stack gas of utility generating stations. Such sorbent-based systems collect integrated samples of mercury from stack gas onto selective sorbent materials over extended time periods (from a few hours to several days). The collected samples are then analyzed for mercury, and the stack gas mercury concentration is calculated. Appendix K defines procedures for use of such systems to collect total vapor-phase mercury in combustion source emissions and requires the use of multi-stage sorbent traps pre-spiked with mercury as a quality assurance (QA) measure. In the test reported here, the HG-324K was verified for measurement of total vapor-phase mercury ( $Hg_T$ ), which is the sum of elemental mercury ( $Hg^0$ ) and oxidized mercury ( $Hg_{OX}$ ) (which is primarily mercuric chloride [ $HgCl_2$ ] vapors). Note that the HG-324K is a sample collection system; the mercury results shown from the HG-324K in this report resulted from use of the HG-324K with sorbent traps prepared and subsequently analyzed for mercury by Frontier Geosciences.

The HG-324K was verified by evaluating the following parameters:

- Relative accuracy (RA)
- Data completeness
- Operational factors such as ease of use, maintenance and data output needs, power and other consumables use, reliability, and operational costs.

The HG-324K was verified during part of a field test that lasted from June 12 to July 25, 2006, and that included two separate four-day periods of reference mercury measurements carried out by ARCADIS Inc., under subcontract to Battelle, using American Society for Testing and Materials (ASTM) D 6784-02, the “Ontario Hydro” (OH) method.<sup>(4)</sup> Specifically, the HG-324K was used to sample stack gas from June 12 through June 15, 2006, and RA was determined by comparing HG-324K vapor-phase mercury results to simultaneous results from 12 two-hour sampling runs with the OH method. Data completeness was assessed as the percentage of maximum data return achieved by the HG-324K over its test period. Operational factors were evaluated by means of operator observations and records of needed maintenance, vendor activities, and expendables use.

The sorbent traps used with the HG-324K for this verification were prepared, and subsequently analyzed for mercury after sampling, by Frontier Geosciences. The traps each contained four separate sections of sorbent. The first section collected mercury from the flue gas; the second collected any breakthrough from the first section; the third was spiked, as required by Appendix K<sup>(1)</sup>, with mercury before sampling; and the fourth collected any mercury lost from the third section during sampling. Trap preparation included spiking the third sorbent section of each trap with nominally 100 ng of mercury. Spike recovery determinations were not based on this nominal value, however. Frontier Geosciences determined the true value of the mercury spike amount as 98.47 ng, by retaining a subset of spiked traps in the laboratory, and determining the amount of mercury on the spiked section of the traps at the same time that the collected samples from this field verification of the HG-324K were analyzed. The mercury analysis by Frontier Geosciences included measurement of mercury on each of the four sorbent stages in each trap, analysis of blank traps, analysis of a mercury Standard Reference Material<sup>®</sup> (National Institute of Standards and Technology [NIST] 1641d), assessment of analytical spike recovery and replicate analysis precision, and analysis of initial and continued calibration blank and continued calibration verification samples.

### 3.2 Test Facility

The host facility for the HG-324K verification was the R.M. Schahfer Generating Station, located near Wheatfield, Indiana, approximately 20 miles south of Valparaiso, Indiana. The Schahfer plant consists of four units (designated 14, 15, 17, and 18), with a total rated capacity of about 1,800 megawatts (MW). The HG-324K was verified at Unit 17, which burns pulverized Illinois sub-bituminous coal and has an electrostatic precipitator and a wet flue gas desulfurization (FGD) unit. Unit 17 has a typical capacity of about 380 MW. The unit was operated near this capacity for most of the test period, although the typical daily pattern of operation was to reduce load substantially for a few hours between late evening and early morning.

Flue gas from Unit 17 feeds into a free-standing concrete chimney with an internal liner. The top of the stack is 499 feet above ground level (agl). Emission test ports and penetrations in the concrete chimney and liner are located at a platform approximately 8 feet wide that encircles the outside of the stack at 370 feet agl. The stack diameter at the platform level is 22 feet 6 inches, so the total flow area is 397.6 square feet. The last flow disturbance is at the FGD connection to the stack liner at 128 feet agl. Thus, the emission test ports were over 10 stack diameters downstream from the last flow disturbance and nearly six diameters upstream from the stack exit. Four emission test ports were located at 90° intervals around the circumference of the stack about 4 feet above the platform at 370 feet agl and were standard 4-inch ports with #125 flanges. No traversing was done during sampling; both the OH method and the HG-324K sampled from a single fixed point one meter inside the inner liner of the stack at their respective port locations. This arrangement was justified by the absence of stratification observed for sulfur dioxide (SO<sub>2</sub>) and nitrogen oxides (NO<sub>x</sub>) at this sampling location.

Table 3-1 summarizes key operating and stack gas conditions that characterize Schahfer Unit 17 during the field period, showing the range and average values of key parameters and constituents. Stack gas pressure was slightly positive at the sampling location.

**Table 3-1. Operating and Stack Gas Conditions at Schahfer Station Unit 17**

Parameter	Average	Range
Unit 17 Load <sup>a</sup>	334 MW	140–391
Coal Feed Rate <sup>a</sup>	297 klb/hr <sup>c</sup>	140–374
Temperature <sup>a</sup>	130°F	118–140
Moisture <sup>b</sup>	14.8 %	13.3–15.3
NO <sub>x</sub> <sup>a</sup>	97 ppm <sup>d</sup>	61–165
SO <sub>2</sub> <sup>a</sup>	193 ppm	104–316
Total mercury vapor <sup>b</sup>	0.81 µg/dscm <sup>e</sup>	0.73–0.93

a: Values calculated from hourly data recorded by R.M. Schahfer staff June 12 to July 25, 2006.

b: Values based on measurements made during OH reference sampling periods June 12–15, 2006.

c: klb/hr = thousands of pounds per hour.

d: ppm = parts per million.

e: µg/dscm = micrograms per dry standard cubic meter.

### 3.3 Test Procedures

Following are the test procedures used to evaluate the HG-324K.

#### 3.3.1 Relative Accuracy

The RA of the HG-324K was evaluated by comparing its Hg<sub>T</sub> results to simultaneous results obtained by sampling stack gas with the OH method. The OH method is the currently accepted reference method for mercury measurements in stack gas, and employs dual impinger trains sampling in parallel through a common probe to determine oxidized and elemental vapor-phase mercury by means of appropriate chemical reagents.<sup>(4)</sup> Over the period of June 12 to 15, ARCADIS conducted a series of 12 OH runs on the Unit 17 stack, each two hours in duration, using paired OH trains. The Hg<sub>T</sub> concentration determined by the OH reference method in each run was compared to the corresponding result from paired HG-324K traps sampled over exactly the same time period as the OH run.

The OH trains were dismantled for sample recovery in the field by ARCADIS staff, and all collected sample fractions were logged and stored for transfer to the ARCADIS analytical laboratory. All sample handling, quality assurance/quality (QA/QC) activities, and mercury analyses were conducted by ARCADIS. Subsequent to mercury analysis, ARCADIS reviewed the data and reported final mercury results from all trains in units of µg/dscm. The results from the paired OH trains were checked relative to the duplicate precision criterion required of the OH method,<sup>(5)</sup> and qualified OH results were averaged to produce the final reference data. The paired sorbent trap samples collected using the HG-324K were sent to Frontier Geosciences in Seattle, Washington, for mercury analysis. The mercury results from the paired HG-324K sorbent traps were reviewed for spike recovery and duplicate precision relative to Appendix K requirements.<sup>(1)</sup> RA was calculated as described in Section 5.1, and in addition the average of all HG-324K results was compared to the average of all OH results.



### 3.3.2 Data Completeness

No additional test procedures were carried out specifically to address data completeness of the HG-324K. This parameter was assessed by comparing the overall data return to the total possible data return.

### 3.3.3 Operational Factors

Operational factors such as maintenance needs, data output, consumables use, and ease of use were evaluated based on observations by Battelle and Schahfer facility staff. Examples of information used to assess operational factors were the use or replacement of any consumables, the effort or cost associated with maintenance or repair, vendor effort (e.g., time on site) for repair or maintenance, the duration and causes of any down time or data acquisition failure, and observations about ease of use of the HG-324K.

### 3.4 Verification Schedule

The HG-324K was verified in a field effort that took place from June 12 to July 25, 2006, that also evaluated two mercury CEMs and one other sorbent-based system. The HG-324K was installed at the Unit 17 stack on June 11 and removed on June 16, 2006, during which period it was operated by a vendor representative. Twelve successive OH reference method runs were carried out in this period for comparison to the HG-324K results.

Table 3-2 shows the actual schedule of OH reference method sampling completed by ARCADIS between June 12 and 15, 2006. The OH sampling proceeded efficiently, with three runs conducted on each of four successive days. In all cases, the HG-324K vendor representative was informed of the planned start time of each OH run; and, in a few instances, the start time of a run was delayed slightly to assure that the technologies being tested were fully ready to obtain data during the OH run. All OH runs were of exactly two hours duration.

**Table 3-2. Schedule of OH Method Sampling in the Week of June 12, 2006**

Run Number	Date	Start Time	End Time
1	6/12/06	09:15	11:15
2	6/12/06	12:15	14:15
3	6/12/06	15:40	17:40
4	6/13/06	08:15	10:15
5	6/13/06	11:10	13:10
6	6/13/06	14:05	16:05
7	6/14/06	08:10	10:10
8	6/14/06	11:25	13:25
9	6/14/06	14:30	16:30
10	6/15/06	08:20	10:20
11	6/15/06	11:05	13:05
12	6/15/06	13:45	15:45

Following the field sampling effort, all HG-324K sorbent trap samples were shipped by Environmental Supply Company to Frontier Geosciences for analysis. Frontier Geosciences returned an analysis data file that included results of blank, replicate analysis, and other QA/QC results, along with the calculated stack gas mercury concentrations from each sorbent trap both uncorrected and corrected for mercury spike recovery.

## Chapter 4 Quality Assurance/Quality Control

QA/QC procedures were performed in accordance with the quality management plan (QMP) for the AMS Center<sup>(6)</sup> and the test/QA plan for this verification test.<sup>(3)</sup> QA/QC procedures and results are described below.

### 4.1 OH Reference Method

This verification test included a comparison of HG-324K results to those of the OH reference method for flue gas mercury.<sup>(4)</sup> The quality of the reference measurements was assured by adherence to the requirements of the OH method, including requirements for solution and field blanks, spiked samples, and initial and continuing blanks and calibration standards. In addition, all OH reference measurements were made with paired trains, and the percent relative deviation (%RD) of each data pair was required to be  $\leq 10\%$  (at mercury levels  $>1.0 \mu\text{g/dscm}$ ) or  $\leq 20\%$  (at mercury levels  $\leq 1.0 \mu\text{g/dscm}$ ) (%RD = difference between the paired train results divided by sum of those results, expressed as a percentage).<sup>(5)</sup> The following sections present key data quality results from the OH method.

#### 4.1.1 OH Reproducibility

The mercury results of the OH stack gas samples are shown in Table 4-1 for the June 12 to 15 period of OH method sampling. The table indicates the OH run number, and lists the average vapor phase  $\text{Hg}_{\text{OX}}$ ,  $\text{Hg}^0$ , and  $\text{Hg}_{\text{T}}$  results from the paired OH trains in each run, and the percent relative deviation of each pair of results. All mercury results are in micrograms of mercury per dry standard cubic meter ( $\mu\text{g/dscm}$ ).

Inspection of Table 4-1 shows that  $\text{Hg}_{\text{T}}$  in the Unit 17 stack ranged from 0.73 to 0.93  $\mu\text{g/dscm}$  in the OH runs conducted in the June 12–15 period. The average  $\text{Hg}_{\text{T}}$  value was 0.81  $\mu\text{g/dscm}$ .  $\text{Hg}^0$  comprised the great majority of the  $\text{Hg}_{\text{T}}$ , consistent with the scrubbing of the Schahfer Unit 17 flue gas.  $\text{Hg}_{\text{OX}}$  never exceeded about 0.07  $\mu\text{g/dscm}$  and was typically about 5% of the  $\text{Hg}_{\text{T}}$ .

Table 4-1 shows close agreement between the paired OH train results for all three mercury fractions. The %RD values in Table 4-1 are less than about 5% in all 12 runs for both  $\text{Hg}^0$  and  $\text{Hg}_{\text{T}}$ . The %RD values for the relatively very low  $\text{Hg}_{\text{OX}}$  concentrations are slightly higher, with

**Table 4-1. OH Results from June 12–15, 2006, Sampling Period**

OH Run	Average Mercury Concentration ( $\mu\text{g}/\text{dscm}$ ) and %RD of Paired Train Results <sup>(a)</sup>					
	Hg <sub>OX</sub>	%RD	Hg <sup>0</sup>	%RD	Hg <sub>T</sub>	%RD
1	0.022	15.3	0.762	3.6	0.783	3.0
2	0.037	6.8	0.822	3.8	0.859	3.4
3	0.038	3.9	0.821	1.1	0.859	0.9
4	0.058	3.4	0.875	2.0	0.933	1.7
5	0.053	6.6	0.795	0.6	0.848	0.1
6	0.048	11.4	0.684	4.9	0.732	5.3
7	0.072	1.2	0.739	2.1	0.811	2.0
8	0.060	0.5	0.690	4.3	0.750	3.9
9	0.055	5.0	0.819	1.9	0.874	1.5
10	0.054	0.2	0.766	3.9	0.820	3.6
11	0.037	2.5	0.691	1.1	0.727	0.9
12	0.032	1.8	0.748	2.4	0.781	2.4

<sup>(a)</sup> %RD = difference between paired train results divided by sum of paired train results.

two values exceeding 10%. The applicable acceptance criterion for all the paired OH results is %RD < 20%, because all OH mercury results from this set of OH runs were less than 1  $\mu\text{g}/\text{dscm}$ .<sup>(5)</sup> All results in Table 4-1 met that criterion, even for the Hg<sub>OX</sub> fraction, which was present at very low concentrations.

#### 4.1.2 OH Blank and Spike Results

Analyses were conducted on eight total samples collected at the Schahfer site from the blank reagents used in the OH method between June 12 and 15. Only two of those samples showed detectable mercury, with concentrations of 0.004  $\mu\text{g}/\text{L}$ . This blank reagent concentration is negligible compared to the mercury in impinger solutions recovered from trains after stack sampling. Those recovered sample concentrations were typically about 0.1  $\mu\text{g}/\text{L}$ , 0.2  $\mu\text{g}/\text{L}$ , and 3  $\mu\text{g}/\text{L}$  in potassium chloride (KCl) solution, hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) solution, and potassium permanganate (KMnO<sub>4</sub>) solution, respectively.

Blank OH sampling trains were prepared and taken to the sampling location on the Unit 17 stack on three occasions in the period of OH sampling and were then returned for sample recovery without exposure to stack gas. These blank OH trains provide additional assurance of the quality of the train preparation and recovery steps. For the June 12 to 15 sampling period, the total amounts of mercury recovered from the three blank trains range from 0.126 to 0.144  $\mu\text{g}$ , equivalent to approximately 7% of the typical total amount of mercury recovered from a train after stack sampling at the Schahfer plant. Those blank train results correspond to stack gas mercury concentrations of less than 0.06  $\mu\text{g}/\text{dscm}$  under typical sampling conditions in this verification.

All initial and continuing blank and calibration values from laboratory analysis of the OH samples met the requirements of the OH method. The recovery of mercury spiked into each reagent solution recovered from blank and sampled OH trains was also evaluated during laboratory analysis. Those spike recoveries ranged from 85 to 112%, and averaged 93%. The recovery of mercury spiked into blank train samples as part of the performance evaluation (PE) audit also met the prescribed criteria, as described in Section 4.2.1.

## 4.2 Audits

Three types of audits were performed during the verification test: a PE audit of the OH reference method, a technical systems audit (TSA) of the verification test performance, and a data quality audit. Audit procedures are described further below.

### 4.2.1 Performance Evaluation Audits

PE audits of the OH method were carried out through procedures implemented at the Schahfer plant during the field period. Table 4-2 summarizes the procedures and results of the PE audits of the OH reference method, showing the parameter audited, the date of the audit, the OH and reference values, the observed agreement, and the target agreement. The OH method incorporates dual sampling trains, and the equipment used by ARCADIS to carry out the OH sampling included dual Model 522 Source Sampler meter boxes (Apex Instruments, Fuquay-Varina, North Carolina) designated by serial number as #2007 and #2008. As a result, for some parameters, Table 4-2 includes results for both meter boxes or for both of the dual OH trains.

Four PE audits were conducted:

- A Fluke Model 52 II digital thermometer (Serial No. 80730162) was used to audit the probe temperature measurements made by the #2007 meter box and the stack temperature measurements made by the #2008 meter box. For this comparison, the appropriate thermocouple was disconnected from the meter box and connected to the Fluke thermometer.
- A BIOS International Corporation DryCal NIST-traceable flow measurement standard (Model DC2-B, Serial No. 103777, vendor-calibrated on May 9, 2006) was used to audit the sample gas flow rate with each of the two OH meter boxes. Note that this audit was conducted during a second period of OH sampling carried out in this verification test in July 2006, rather than in the June 12 to 15 period used for verification of the HG-324K.
- A set of weights (Rice Lake Weight Set, Serial No. 1JXA) calibrated to ASTM Class 3 standards was used to audit the electronic balance (AND FP-6000, Serial No. 6402118) used for weighing the OH method impingers.
- Recovery of mercury from OH trains was audited by spiking impingers containing KCl, H<sub>2</sub>O<sub>2</sub>/nitric acid (HNO<sub>3</sub>), and KMnO<sub>4</sub>/sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) reagents in two blank OH impinger trains, with 1 milliliter (mL) of a prepared mercury solution, in each of the two separate periods of OH sampling. The mercury spiking solution was 2.5 µg/mL Hg in 1% HNO<sub>3</sub> and was prepared by dilution of a NIST-traceable 1,000-ppm (i.e., 1,000-µg/mL) standard (Aa34n-1, Accustandards, Inc.). In the first week of OH sampling, Impingers 2, 4, and 5 of Blank Trains 8L and 8R were spiked; and, in the final week of OH sampling, Impingers 2, 4, and 6 of Blank Trains 7L and 7R were spiked.

Table 4-2 shows that all the PE audit results were within the target tolerances set in the test/QA plan.<sup>(3)</sup>

**Table 4-2. Summary of PE Audit Results**

Parameter	Date	OH Result	Reference Value	Observed Agreement	Target Agreement	
OH temperature measurement	6/14/06 probe T stack T	228°F <sup>(a)</sup>	230°F	0.29%	2% absolute T	
		127°F <sup>(b)</sup>	129°F	0.31%		
OH sample flow measurement	7/11/06	15.02 L/min <sup>(a)</sup>	14.56 L/min	3.2%	5%	
		14.58 L/min <sup>(b)</sup>	14.35 L/min	1.6%		
Impinger weighing	6/14/06	199.72	200 grams	0.14%	Greater of 1% or 0.5 gram	
		499.27	500 grams	0.15%		
Mercury spike recovery	6/14/06 train 8L	imp 2	2.48 µg	2.5 µg	0.8%	25%
		imp 4	2.02 µg	2.5 µg	19.2%	25%
		imp 5	2.08 µg	2.5 µg	16.8%	25%
	train 8R	imp 2	2.47 µg	2.5 µg	1.2%	25%
		imp 4	1.97 µg	2.5 µg	21.2%	25%
		imp 5	2.10 µg	2.5 µg	16.0%	25%
	7/12/06 train 7L	imp 2	2.24 µg	2.5 µg	10.4%	25%
		imp 4	2.12 µg	2.5 µg	15.2%	25%
		imp 6	2.38 µg	2.5 µg	4.8%	25%
	train 7R	imp 2	2.27 µg	2.5 µg	9.2%	25%
		imp 4	2.33 µg	2.5 µg	6.8%	25%
		imp 6	2.39 µg	2.5 µg	4.4%	25%

<sup>(a)</sup> #2007 meter box.

<sup>(b)</sup> #2008 meter box.

L/min = liters per minute; T = temperature; imp = impinger.

#### 4.2.2 Technical Systems Audit

A Battelle Quality Management representative conducted a TSA at the Schahfer test site on June 14 to ensure that the verification test was being conducted in accordance with the test/QA plan<sup>(3)</sup> and the AMS Center QMP.<sup>(6)</sup> As part of the TSA, test procedures were compared to those specified in the test/QA plan,<sup>(3)</sup> and data acquisition and handling procedures, as well as the reference standards and method were reviewed. The Quality Management representative observed OH method sampling and sample recovery processes, interviewed ARCADIS personnel, and observed the PE audit procedures noted above, except for the OH sample flow and second OH train spiking audits, which were conducted at a later date. Observations and findings from the TSA were documented and submitted to the Battelle Verification Test Coordinator for response. None of the findings of the TSA at the Schahfer site required corrective action. In addition, an internal TSA was conducted in the laboratory charged with analyzing the OH samples. This TSA was conducted by the ARCADIS independent QA Officer in the laboratory on-site at EPA in Research Triangle Park, North Carolina, on July 19 and July 27, 2006. None of the findings of this laboratory TSA required corrective action. Records from both TSA efforts are permanently stored with the Battelle Quality Manager.

#### **4.2.3 Data Quality Audit**

At least 10% of the data acquired during the verification test were audited. Battelle's Quality Manager traced the data from the initial acquisition, through reduction and statistical analysis, to final reporting to ensure the integrity of the reported results. All calculations performed on the data undergoing the audit were checked.

#### **4.3 QA/QC Reporting**

Each audit was documented in accordance with Sections 3.3.4 and 3.3.5 of the QMP for the ETV AMS Center.<sup>(6)</sup> Once the audit reports were prepared, the Battelle Verification Test Coordinator ensured that a response was provided for each adverse finding or potential problem and implemented any necessary follow-up corrective action. The Battelle Quality Manager ensured that follow-up corrective action was taken. The results of the TSA were submitted to the EPA.

#### **4.4 Data Review**

Records generated in the verification test received a one-over-one review before these records were used to calculate, evaluate, or report verification results. Data were reviewed by a Battelle technical staff member involved in the verification test. The person performing the review added his/her initials and the date to a hard copy of the record being reviewed.

## Chapter 5 Statistical Methods

The statistical methods used to evaluate the quantitative performance factors listed in Section 3.1 are presented in this chapter. Qualitative observations were also used to evaluate verification test data.

### 5.1 Relative Accuracy

The RA of the HG-324K with respect to the OH reference method results was assessed as a percentage, using Equation 1:

$$RA = \frac{|\bar{d}| + t_{n-1}^{\alpha} \frac{S_d}{\sqrt{n}}}{\bar{x}} \times 100\% \quad (1)$$

where  $d$  refers to the difference between the OH reference mercury concentration and the HG-324K result over the OH sampling period, and  $x$  corresponds to the OH reference mercury concentration.  $S_d$  denotes the sample standard deviation of the differences, while  $t_{n-1}^{\alpha}$  is the  $t$  value for the  $100(1 - \alpha)$ th percentile of the distribution with  $n-1$  degrees of freedom. The RA was determined for an  $\alpha$  value of 0.025 (i.e., 97.5% confidence level, one-tailed). RA was calculated only for total vapor-phase mercury. All paired OH data meeting the method quality criteria were eligible for inclusion in the calculation of RA. All 12 OH runs met the quality criteria and were included in the RA calculation for the HG-324K. A RA of less than 20% is considered acceptable.<sup>(1)</sup> Alternatively, when the mean reference mercury level is less than 5.0  $\mu\text{g}/\text{dscm}$  (as in this test), agreement of the overall average HG-324K value within 1.0  $\mu\text{g}/\text{dscm}$  of the mean OH value is also considered acceptable.<sup>(1)</sup>

### 5.2 Data Completeness

Data completeness was calculated as the percentage of the total possible data return that was achieved by the HG-324K over its several days of operation in the field. The primary form of data completeness was the number of OH runs (out of 12) for which HG-324K produced valid data. In addition, any down time when the HG-324K would not have been available to carry out a measurement was judged as incomplete data. The causes of any substantial incompleteness of data were established from operator observations or vendor records.



## Chapter 6 Test Results

The results of the verification tests of the HG-324K sorbent-based sampling system are presented below for each of the performance parameters.

### 6.1 Relative Accuracy

The RA of the HG-324K with respect to the OH results for  $Hg_T$  was calculated using Equation 1 in Chapter 5. The primary calculation of RA was conducted using the data from all collected HG-324K sorbent samples. In addition, RA was calculated after applying the acceptance criteria and spike recovery correction required under Appendix K<sup>(1)</sup> to the HG-324K sorbent trap results. These additional calculations were made to illustrate the impact on RA results if these criteria were applied.

Table 6-1 summarizes the results obtained from the HG-324K sorbent sampling system. Table 6-1 lists the date, run number, and trap number of each HG-324K sorbent sample; the blank-corrected  $Hg_T$  concentration in stack gas determined by each of the sorbent traps; and the corresponding average concentration of each pair of traps. Also shown are the spike recovery percentage found for each trap; the  $Hg_T$  concentration that results from applying the spike recovery correction to each trap as indicated in Appendix K;<sup>(1)</sup> the corresponding average spike-corrected concentration of each pair of traps; and the percent relative difference of the spike-corrected paired trap results.

Table 6-1 shows that the  $Hg_T$  results from paired sorbent traps were generally closely similar, with the exception of the results from Run 8. In that run the HG-324K post-test leak check failed, and thus the results from Run 8 are excluded from comparison to the OH results. Table 6-1 also shows the spike recovery percentage for each trap, and indicates that this percentage was outside the acceptable range of 75 to 125% for three traps. The results from those traps are also excluded from comparisons of the spike-corrected HG-324K results to the OH results, in Section 6.1.2. It is noteworthy that those paired traps exhibiting substantial differences in mercury spike recovery did not exhibit comparable differences in the measured  $Hg_T$  concentration in the stack before correction for spike recovery (see Runs 1, 3, and 10 in Table 6-1).

The amount of mercury found on the second sorbent section of each HG-324K trap never exceeded 2% of the amount found on the corresponding first sorbent section. As a result, all the HG-324K samples met the 5% mercury breakthrough criterion stated in Appendix K.<sup>(1)</sup>

**Table 6-1. HG-324K Hg<sub>T</sub> Results**

Date/Run/Trap	Hg <sub>T</sub> <sup>a</sup> (µg/dscm)	Pair Avg Hg <sub>T</sub> (µg/dscm)	% Spike Recovery	Spike- Corrected Hg <sub>T</sub> (µg/dscm) <sup>b</sup>	Pair Avg Spike- Corrected Hg <sub>T</sub> (µg/dscm)	%RD <sup>c</sup>
6/12/06 R1 T1	1.045	1.085	67.7%	d	1.209	e
6/12/06 R1 T2	1.125		93.1%	1.209		
6/12/06 R2 T1	1.021	1.049	95.1%	1.074	1.087	1.2%
6/12/06 R2 T2	1.076		97.9%	1.100		
6/12/06 R3 T1	1.071	1.081	64.1%	d	1.081	e
6/12/06 R3 T2	1.092		101.0%	1.081		
6/13/06 R4 T1	1.178	1.215	99.2%	1.188	1.210	1.8%
6/13/06 R4 T2	1.252		101.6%	1.232		
6/13/06 R5 T1	1.082	1.082	102.8%	1.053	1.081	2.6%
6/13/06 R5 T2	1.082		97.6%	1.110		
6/13/06 R6 T1	1.095	1.019	96.3%	1.138	1.036	9.8%
6/13/06 R6 T2	0.943		100.9%	0.934		
6/14/06 R7 T1	0.918	0.906	94.2%	0.974	0.936	4.1%
6/14/06 R7 T2	0.895		99.8%	0.897		
6/14/06 R8 T1	1.453	f				
6/14/06 R8 T2	0.875					
6/14/06 R9 T1	0.962	0.953	87.0%	1.107	1.077	2.8%
6/14/06 R9 T2	0.944		90.2%	1.047		
6/15/06 R10 T1	0.932	0.901	73.8%	d	0.912	e
6/15/06 R10 T2	0.869		95.3%	0.912		
6/15/06 R11 T1	0.793	0.827	79.7%	0.995	1.013	1.8%
6/15/06 R11 T2	0.860		83.4%	1.031		
6/15/06 R12 T1	0.825	0.927	84.9%	0.972	1.063	8.5%
6/15/06 R12 T2	1.029		89.2%	1.154		

a: Results corrected for average blank trap Hg result.

b: Spike-corrected result = (Hg<sub>T</sub>/% Spike Recovery) x 100.

c: %RD (percent relative deviation) = 100 x absolute value of (T1-T2)/(T1+T2).

d: Spike recovery less than 75%, data excluded per Appendix K.

e: Only one valid result, %RD not calculated.

f: Post-check leak test failed, data excluded.

**6.1.1 Relative Accuracy: Uncorrected Data**

Table 6-2 lists the Hg<sub>T</sub> results in µg/dscm from the OH method (see Table 4-1) and the HG-324K (see Table 6-1, third column), for OH runs 1 through 7 and 9 through 12; Run 8 is excluded because of the failed leak test. The RA of the HG-324K based on 11 runs using the uncorrected data is 29.5 %. Also for these 11 runs, the overall average Hg<sub>T</sub> value from the OH reference method is 0.821 µg/dscm, whereas the uncorrected HG-324K average is 1.004 µg/dscm, a difference of 0.183 µg/dscm.

**Table 6-2. Data Used for Comparison of OH and HG-324K Hg<sub>T</sub> Results**

Date	OH Run No. <sup>a</sup>	OH Hg <sub>T</sub> (µg/dscm)	HG-324K Hg <sub>T</sub> (µg/dscm)
6/12/06	1	0.783	1.085
6/12/06	2	0.859	1.049
6/12/06	3	0.859	1.081
6/13/06	4	0.933	1.215
6/13/06	5	0.848	1.082
6/13/06	6	0.732	1.019
6/14/06	7	0.811	0.906
6/14/06	9	0.874	0.953
6/15/06	10	0.820	0.901
6/15/06	11	0.727	0.827
6/15/06	12	0.781	0.927

a: Run 8 excluded from calculation because HG-324K failed post-sampling leak check in that run.

### 6.1.2 Relative Accuracy: Spike-Corrected Data

Table 6-3 lists the Hg<sub>T</sub> results in µg/dscm from the OH method (see Table 4-1) and the spike-corrected results in µg/dscm from the HG-324K (see Table 6-1), for OH runs 1 through 7 and 9 through 12; Run 8 is excluded because of the failed leak test. Table 6-3 also notes which three HG-324K results are from a single trap, as opposed to the average of paired traps, due to low spike recovery on one trap. The RA of the HG-324K based on these 11 runs using the spike-corrected data is 37.0 %. Also for these 11 runs, the overall average Hg<sub>T</sub> value from the OH reference method is 0.821 µg/dscm, whereas the spike-corrected HG-324K average is 1.064 µg/dscm, a difference of 0.243 µg/dscm.

**Table 6-3. Data Used for Comparison of OH and Spike-Corrected HG-324K Hg<sub>T</sub> Results<sup>a</sup>**

Date	OH Run No. <sup>a</sup>	OH Hg <sub>T</sub> (µg/dscm)	Spike-Corrected HG-324K Hg <sub>T</sub> (µg/dscm)
6/12/06	1	0.783	1.209 <sup>b</sup>
6/12/06	2	0.859	1.087
6/12/06	3	0.859	1.081 <sup>b</sup>
6/13/06	4	0.933	1.210
6/13/06	5	0.848	1.081
6/13/06	6	0.732	1.036
6/14/06	7	0.811	0.936
6/14/06	9	0.874	1.077
6/15/06	10	0.820	0.912 <sup>b</sup>
6/15/06	11	0.727	1.013
6/15/06	12	0.781	1.063

a: Run 8 excluded from calculation because HG-324K failed post-sampling leak check in that run.

b: Low spike recovery from one trap; therefore, this result from a single trap; all others from paired traps.

The paired trap precision requirement of <10% RD stated in Appendix K<sup>(2)</sup> was met in the eight valid HG-324K runs in which spike-corrected paired trap results were obtained (see Table 6-1, last column). However, eight runs is below the nine values needed to calculate RA, so that calculation was not done using only the spike-corrected paired trap results.

## **6.2 Data Completeness**

The HG-324K sampled during all 12 of the OH runs conducted June 12-15, 2006, with no delays, breakdowns, or sampling interruptions. All sorbent traps were recovered after sampling, with no broken traps. However, after OH Run 8, the post-test leak check failed; and, as a result, only 11 of the 12 sampling runs (91.7%) were suitable for comparison to the OH reference results.

## **6.3 Operational Factors**

The HG-324K was installed quickly at the Schahfer Unit 17 stack on June 11 and was operated by one vendor representative without serious problems for the subsequent four days of OH reference method sampling. A single failed post-test leak check was the only difficulty encountered over all 12 OH runs. Ease of use was not investigated with a newly trained operator, as the vendor operated the HG-324K during the test period. The sorbent traps obtained from Frontier Geosciences were rugged and uniform in construction, so that no breakage occurred; and no problems were encountered in placing the traps into the end of the sampling probe or recovering them after sampling. The sampling probe used with the HG-324K was simple and relatively light in weight, and was handled by the one vendor operator in all sampling. The HG-324K sorbent sampling system incorporated the usual capabilities of a stack sampling box, but also included data acquisition and transfer capabilities. Those capabilities included wireless communication with a personal computer over distances up to several hundred feet, which allowed review and transfer of the sampling data at any time without interrupting the sampling itself. Data were recorded on magnetic card media in the HG-324K, providing a readily transportable and reliable means of data storage.

## Chapter 7 Performance Summary

The RA of the HG-324K for determining  $Hg_T$  based on 11 OH runs was 29.5%, when the comparison was based on HG-324K results corrected for trap blanks but not corrected for mercury spike recovery. For those 11 runs, the overall average  $Hg_T$  value from the OH reference method was  $0.821 \mu\text{g/dscm}$ , whereas that from the HG-324K was  $1.004 \mu\text{g/dscm}$ , a difference of  $0.183 \mu\text{g/dscm}$ . When comparing HG-324K results corrected for mercury spike recovery, the RA for 11 OH runs was 37.0%, and the OH and HG-324K average values were  $0.821 \mu\text{g/dscm}$  and  $1.064 \mu\text{g/dscm}$ , respectively, a difference of  $0.243 \mu\text{g/dscm}$ .

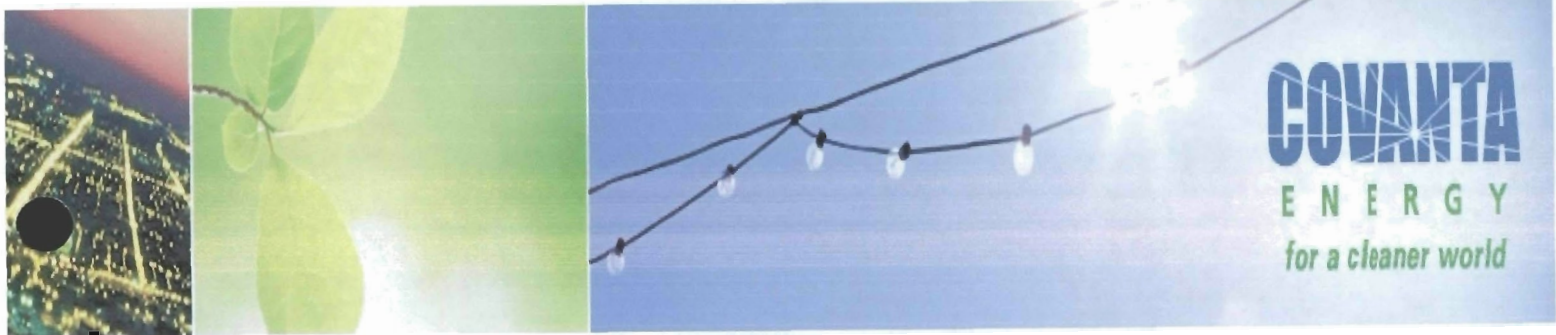
The HG-324K sampled during all 12 OH runs conducted over four days with no delays, breakdowns, broken traps, or sampling interruptions. The only problem encountered was that after Run 8 the post-test leak check failed. As a result, only 11 of the 12 sampling runs (91.7% data completeness) were suitable for comparison to the OH reference results.

The HG-324K was installed quickly and was operated by a vendor representative without serious problems. A failed post-test leak check in one sampling run was the only difficulty encountered. The sorbent traps were rugged and uniform in construction, so that no breakage occurred; no problems were encountered in placing the traps into the end of the sampling probe or recovering them after sampling. The sampling probe used with the HG-324K was simple and relatively light in weight, and was handled by the one vendor operator in all sampling. The HG-324K sorbent sampling system incorporated data acquisition and transfer capabilities, including magnetic card recording media and wireless communication.

The cost of the HG-324K system as tested is \$18,750. The cost of each sorbent trap sample was about \$500, including preparation and pre-spiking of the trap, and analysis for mercury after sampling.

## Chapter 8 References

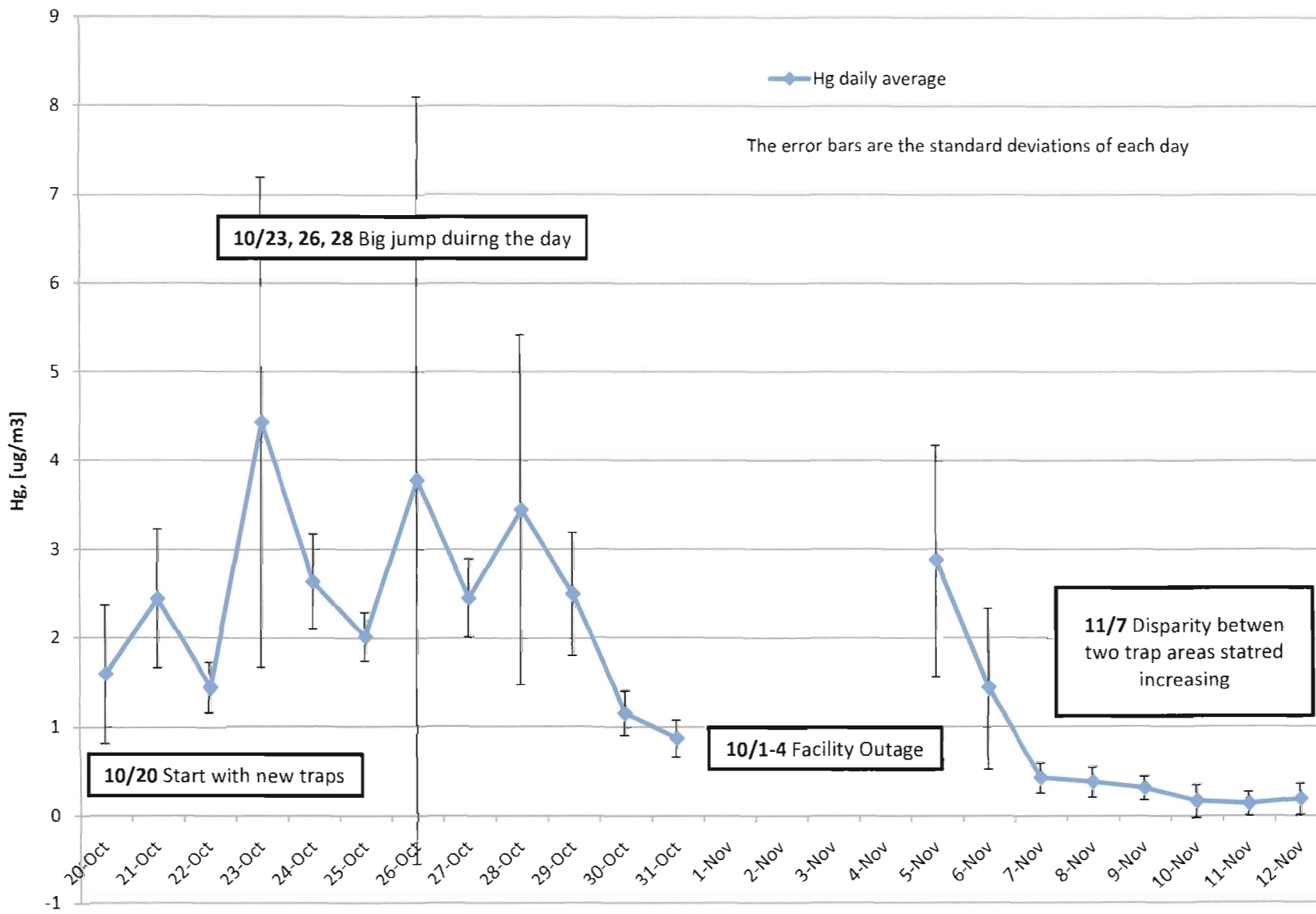
1. *Code of Federal Regulations*, 40 CFR part 75, including Appendices A through K, July 2005.
2. EPA Method 1631, Revision E: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry, EPA-821-R-02-019, August 2002.
3. *Test/QA Plan for Verification of Continuous Emission Monitors and Sorbent-Based Samplers for Mercury at a Coal-Fired Power Plant*, Battelle, Columbus, Ohio, May 18, 2006.
4. *Standard Test Method for Elemental, Oxidized, Particle-Bound and Total Mercury in Flue Gas Generated from Coal-fired Stationary Sources (Ontario Hydro Method)*, ASTM D 6784-02, American Society for Testing and Materials, West Conshohocken, PA, June 2002.
5. *Performance Specification 12A – Specifications and Test Procedures for Total Vapor Phase Mercury Continuous Emission Monitoring Systems in Stationary Sources*, 40 CFR Part 60 Appendix B, July 2005.
6. *Quality Management Plan (QMP) for the ETV Advanced Monitoring Systems Center*, Version 6.0, U.S. EPA Environmental Technology Verification Program, Battelle, Columbus, Ohio, November 2005.



## ATTACHMENT 4

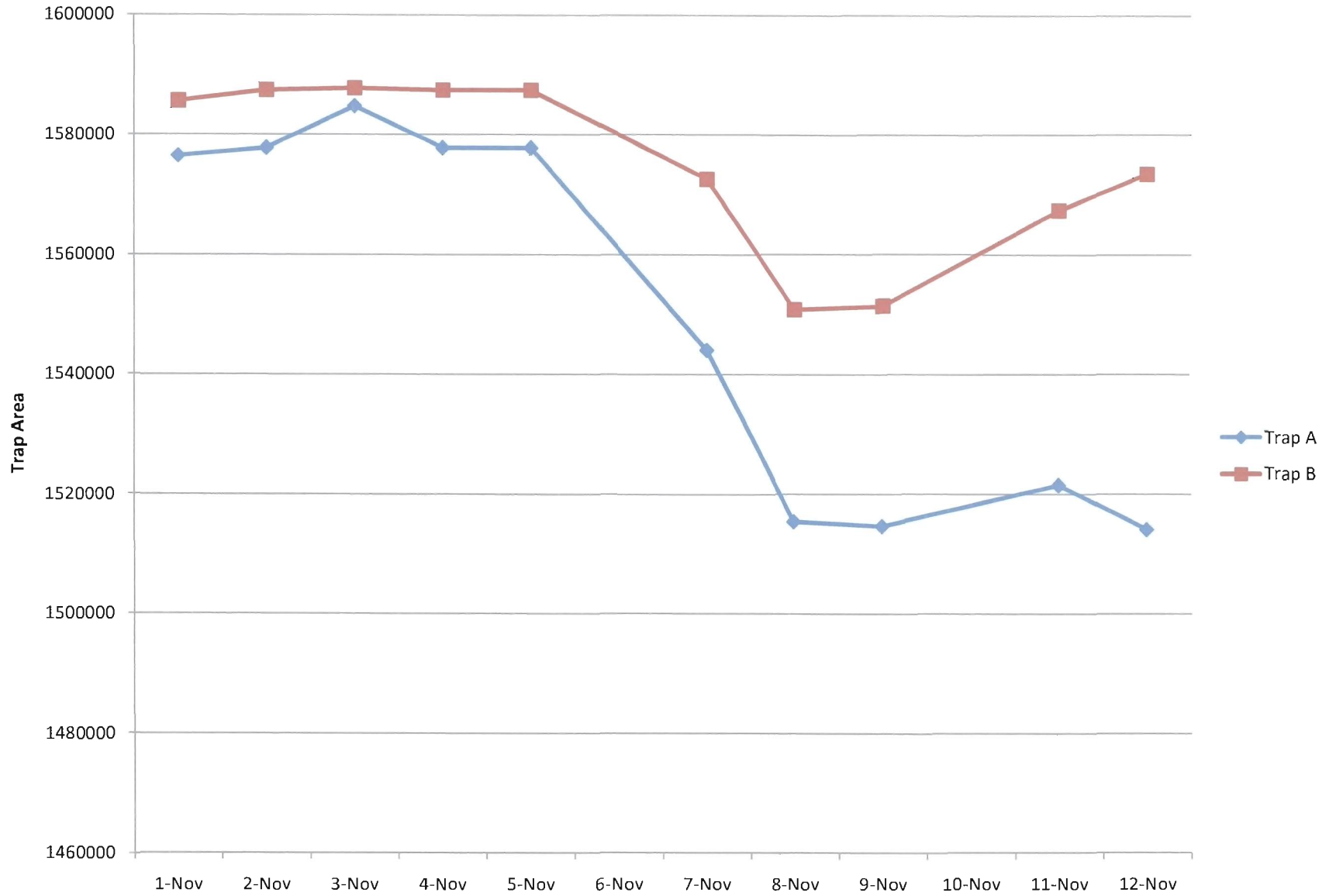
# Analyzer Data and Trends

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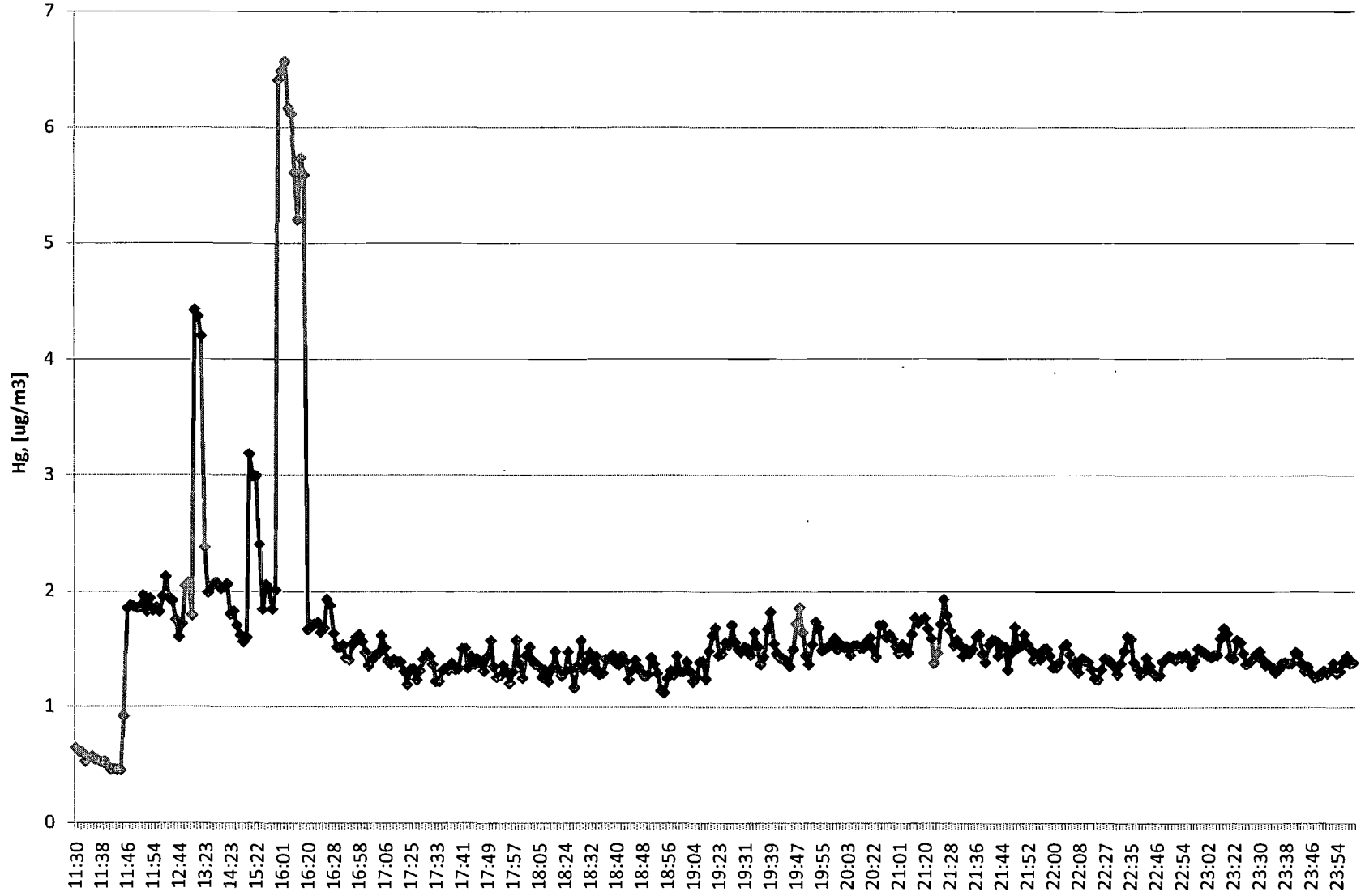




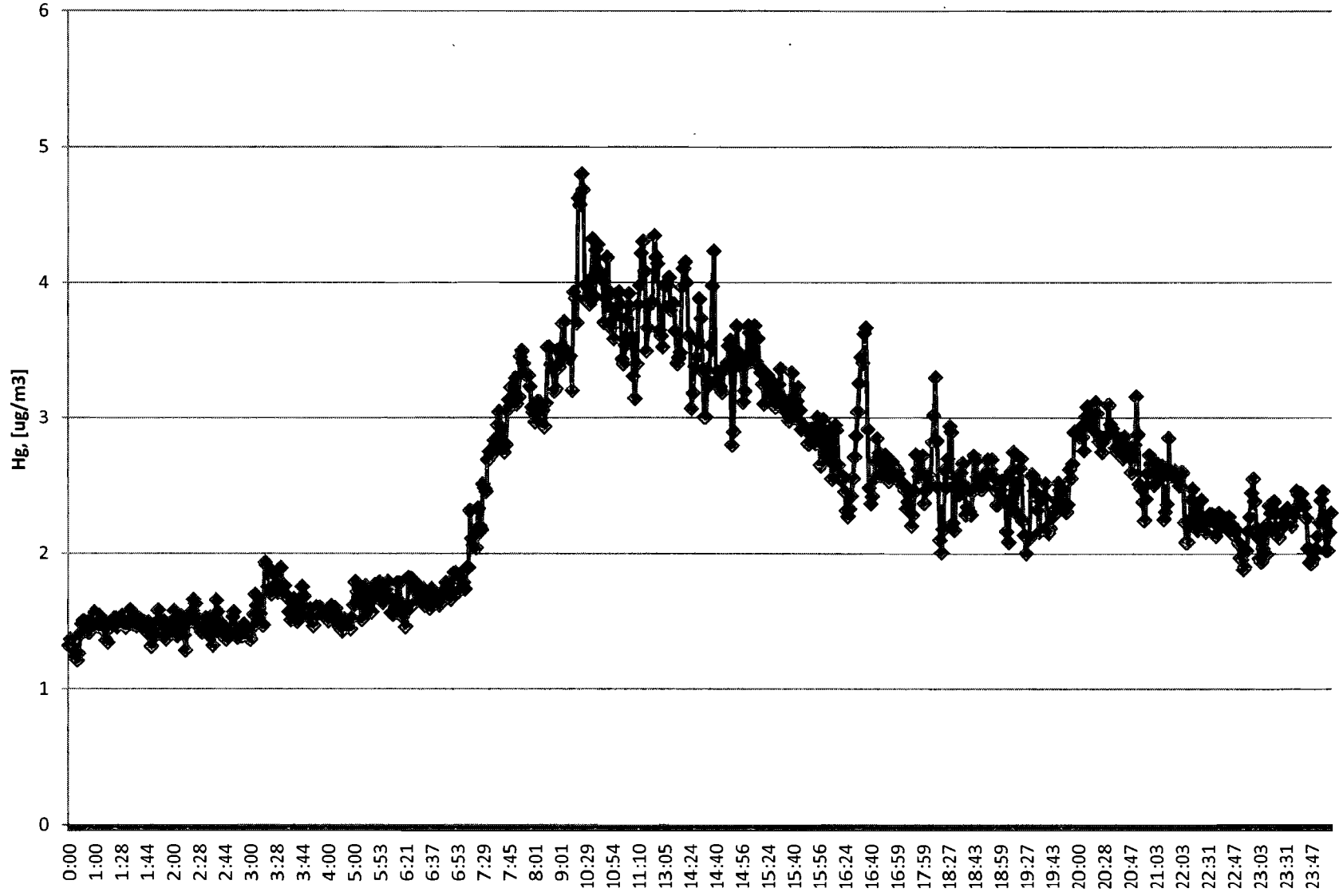
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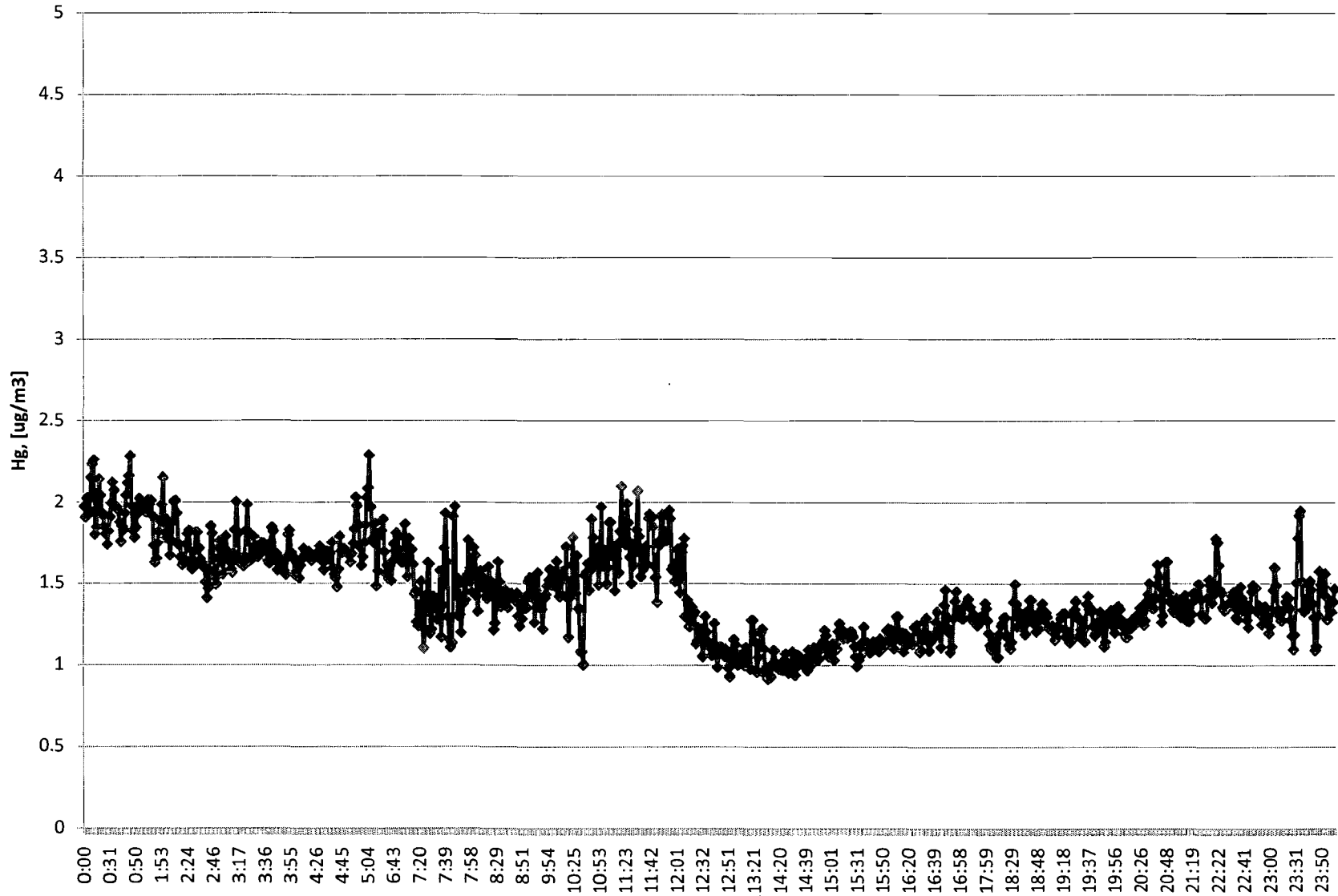
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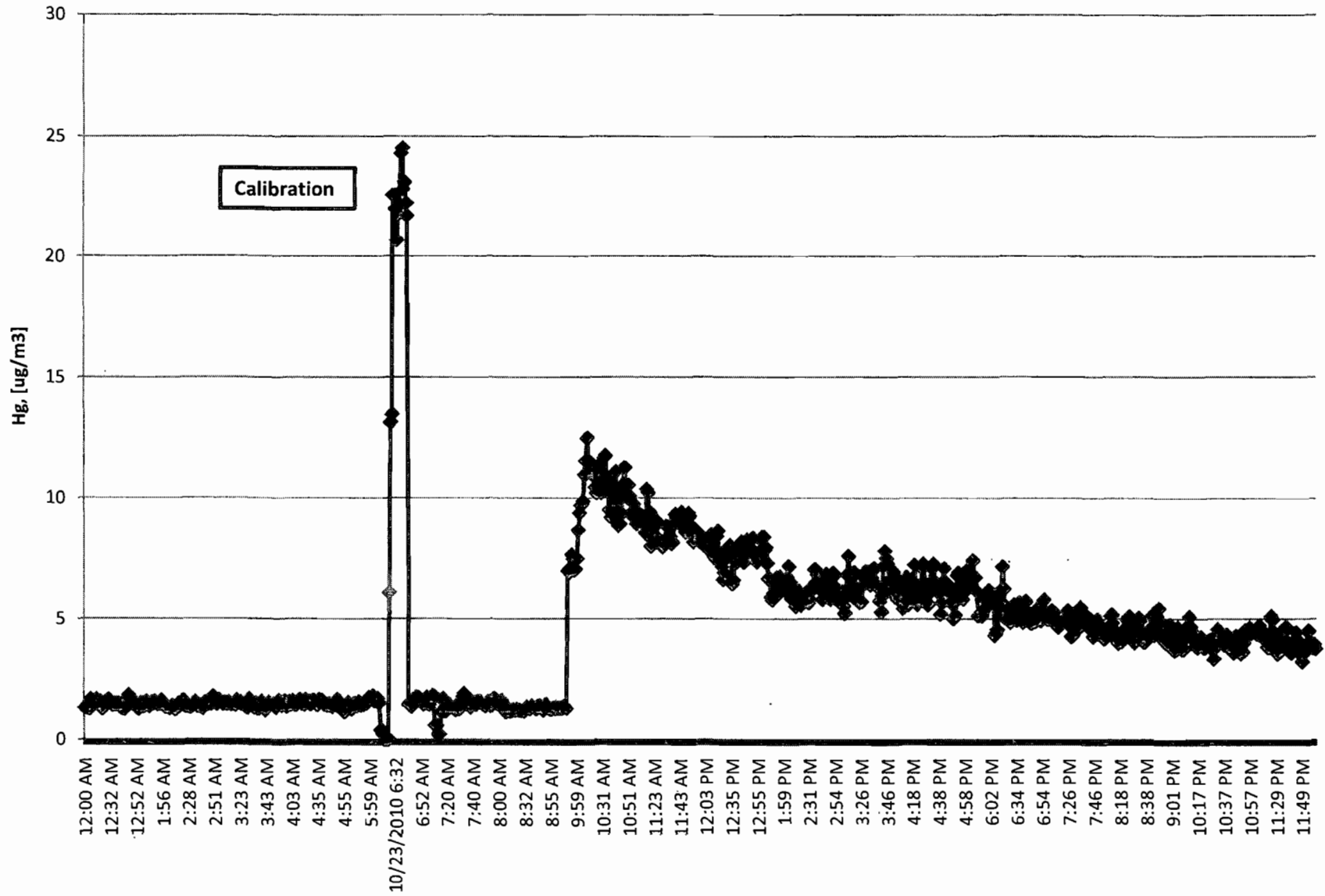
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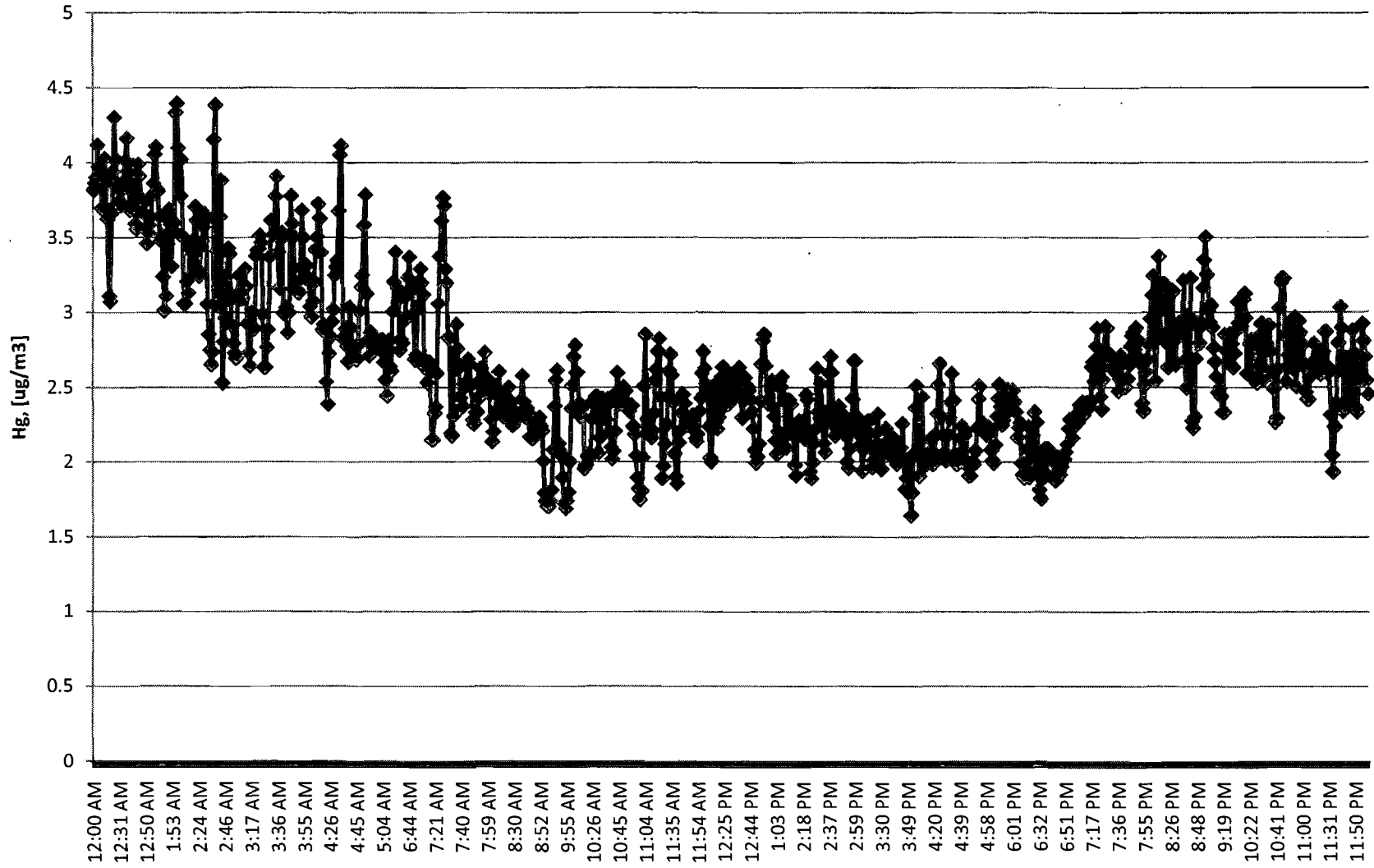
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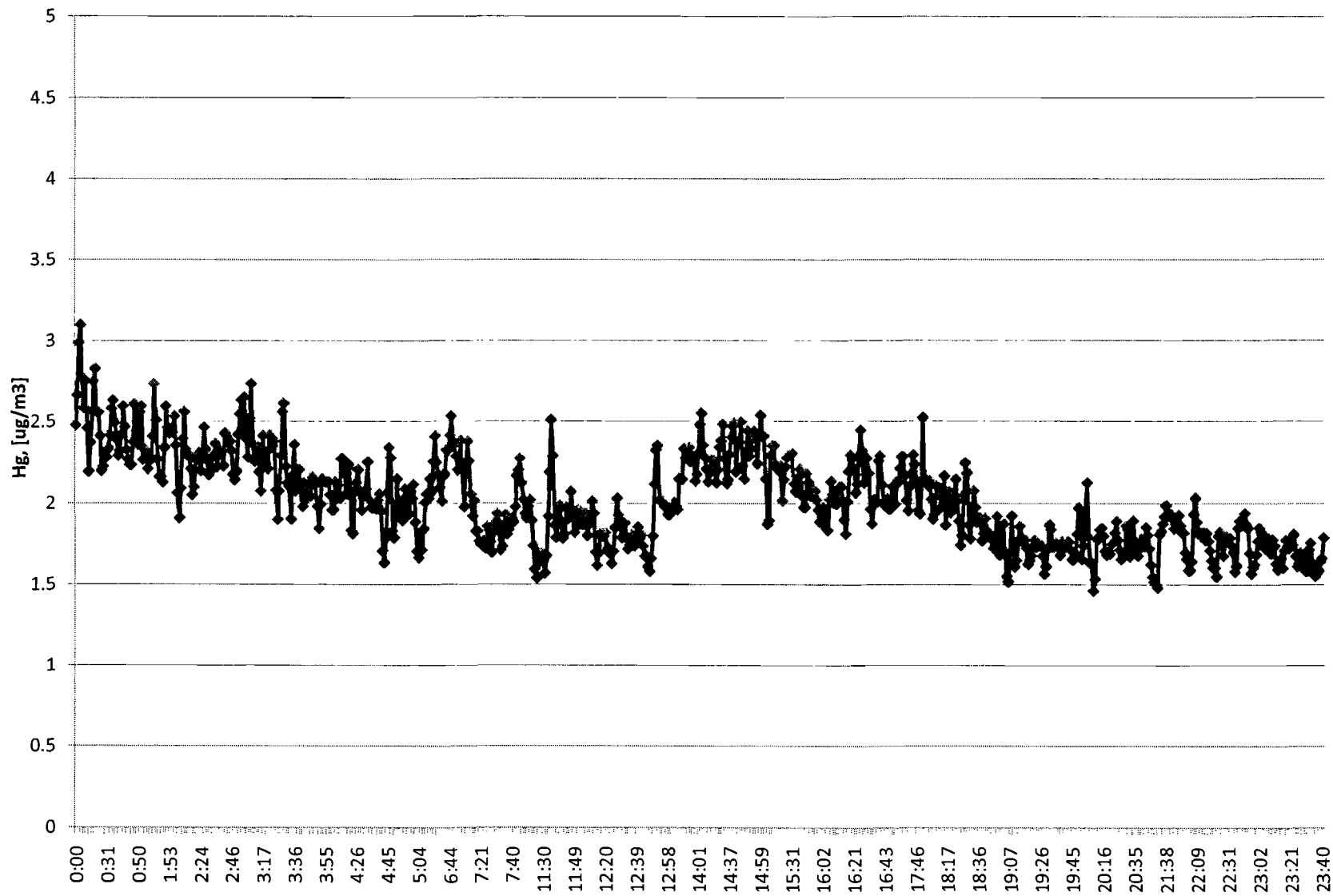
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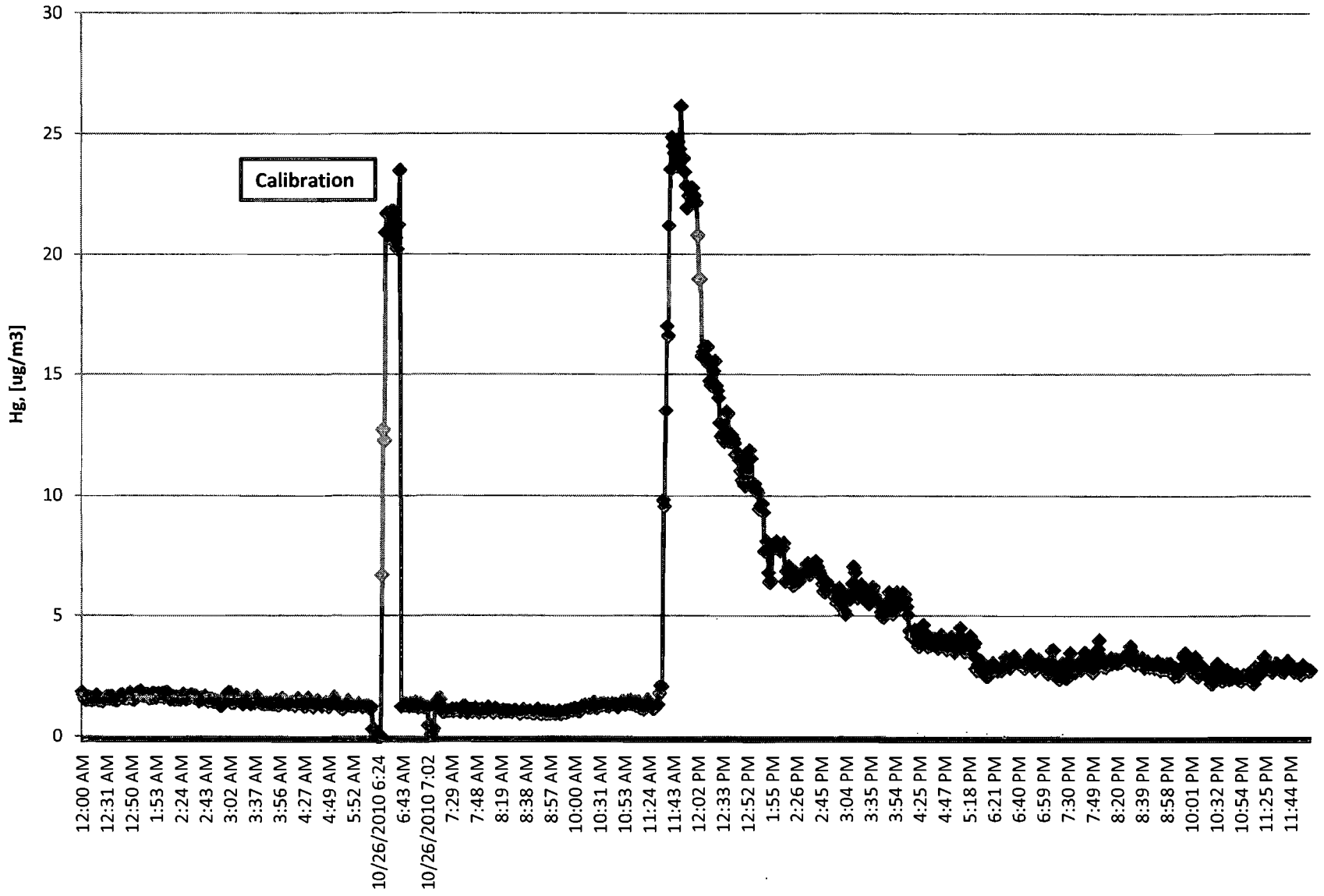
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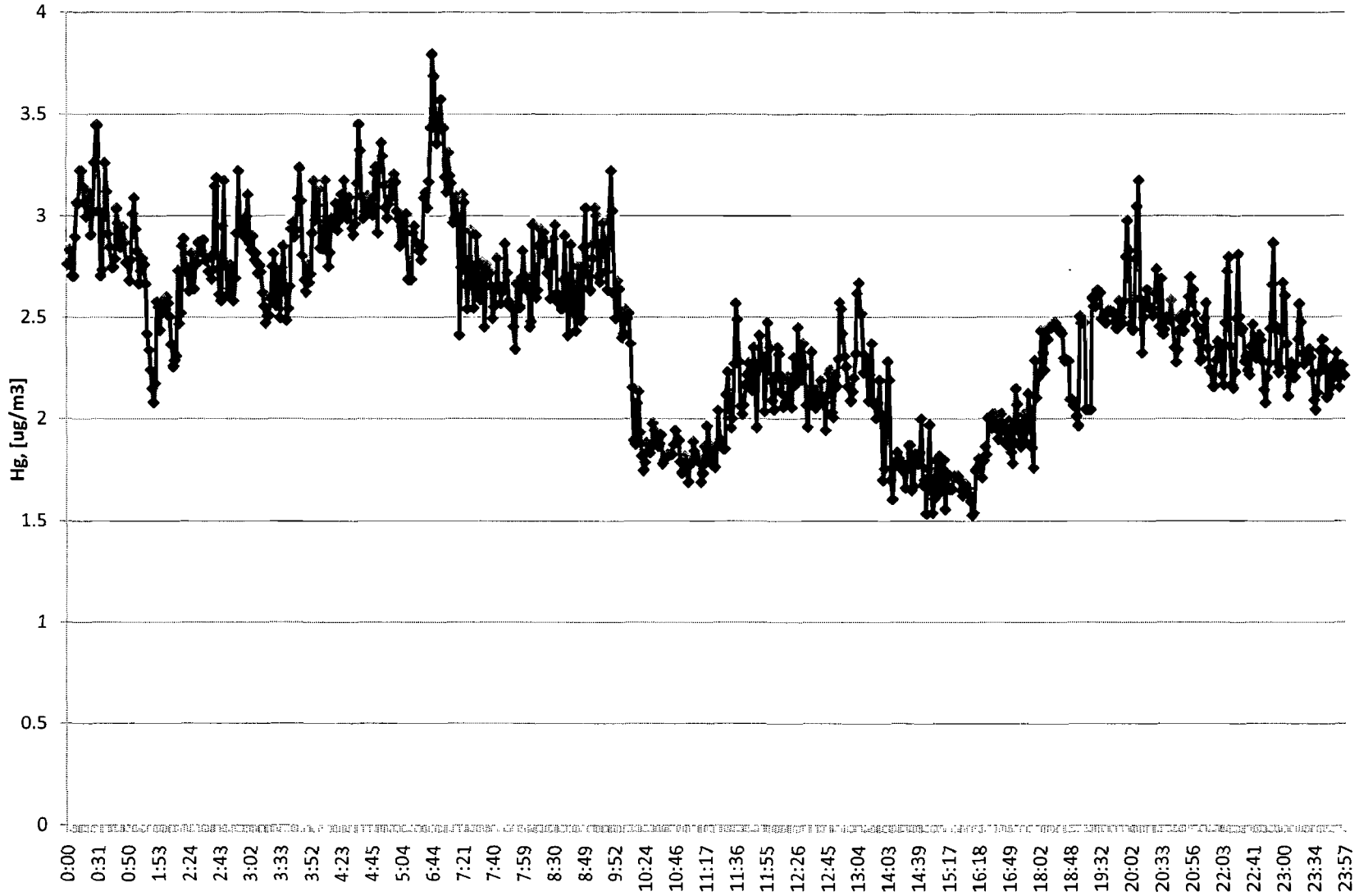


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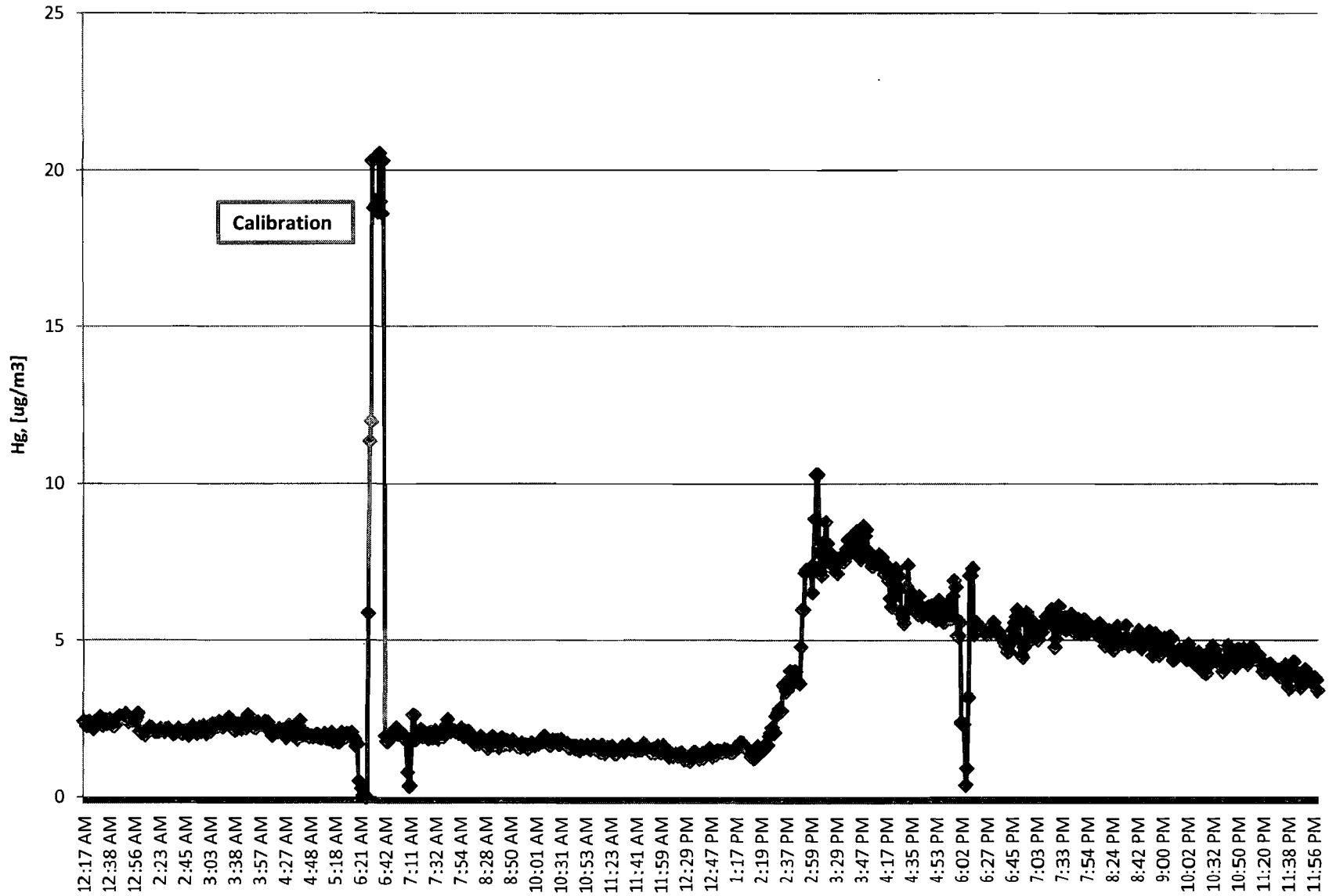




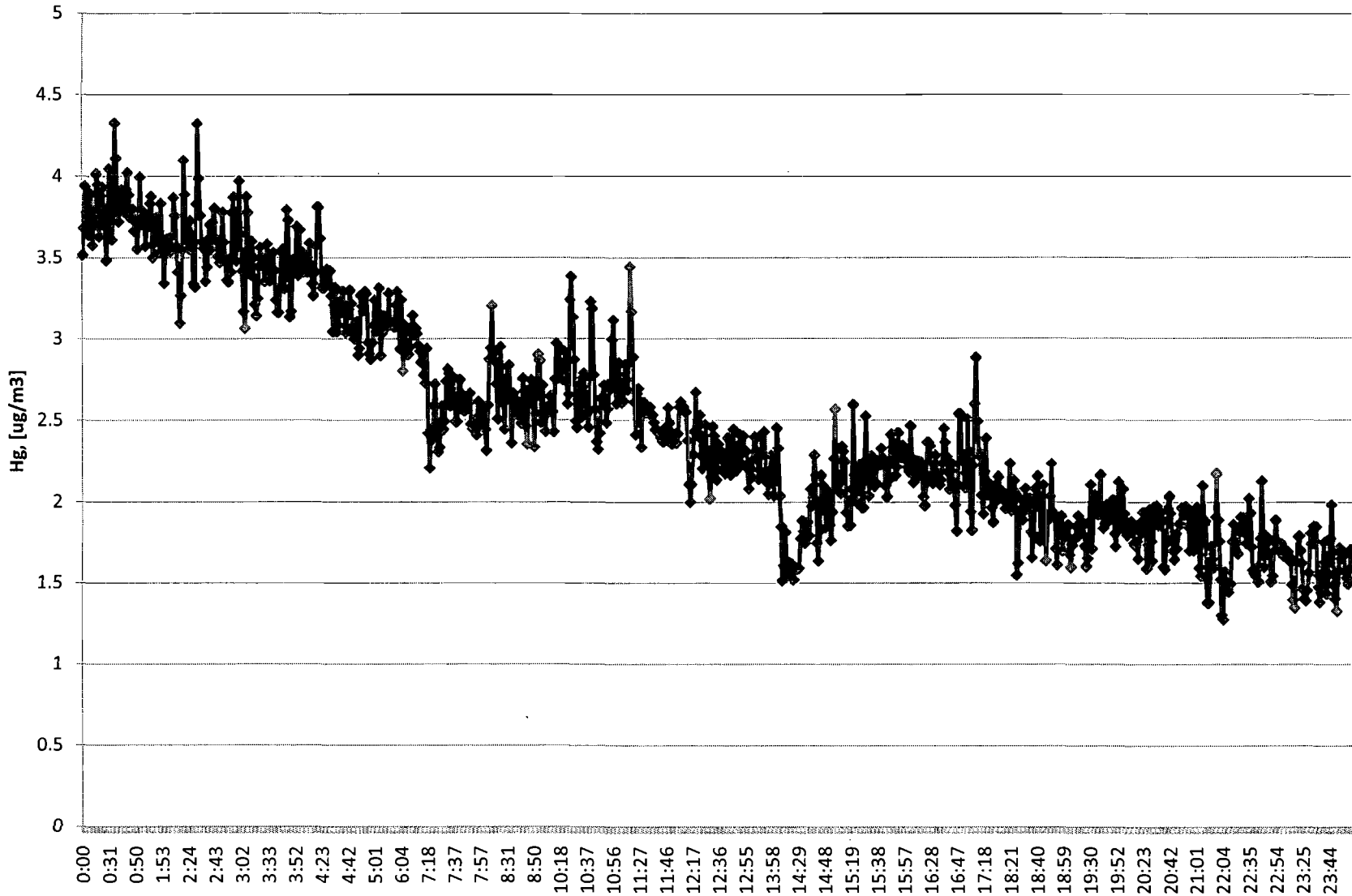
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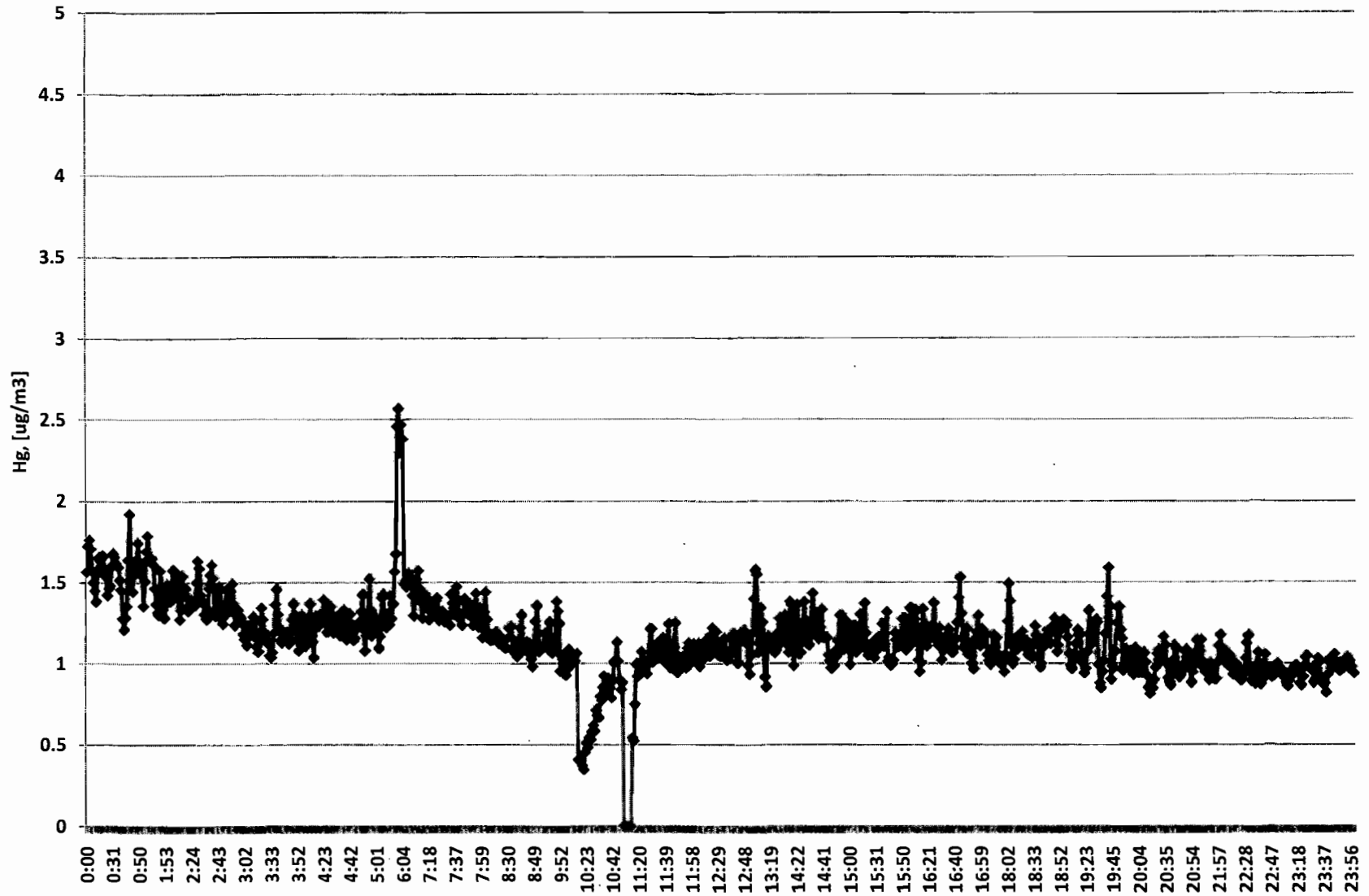
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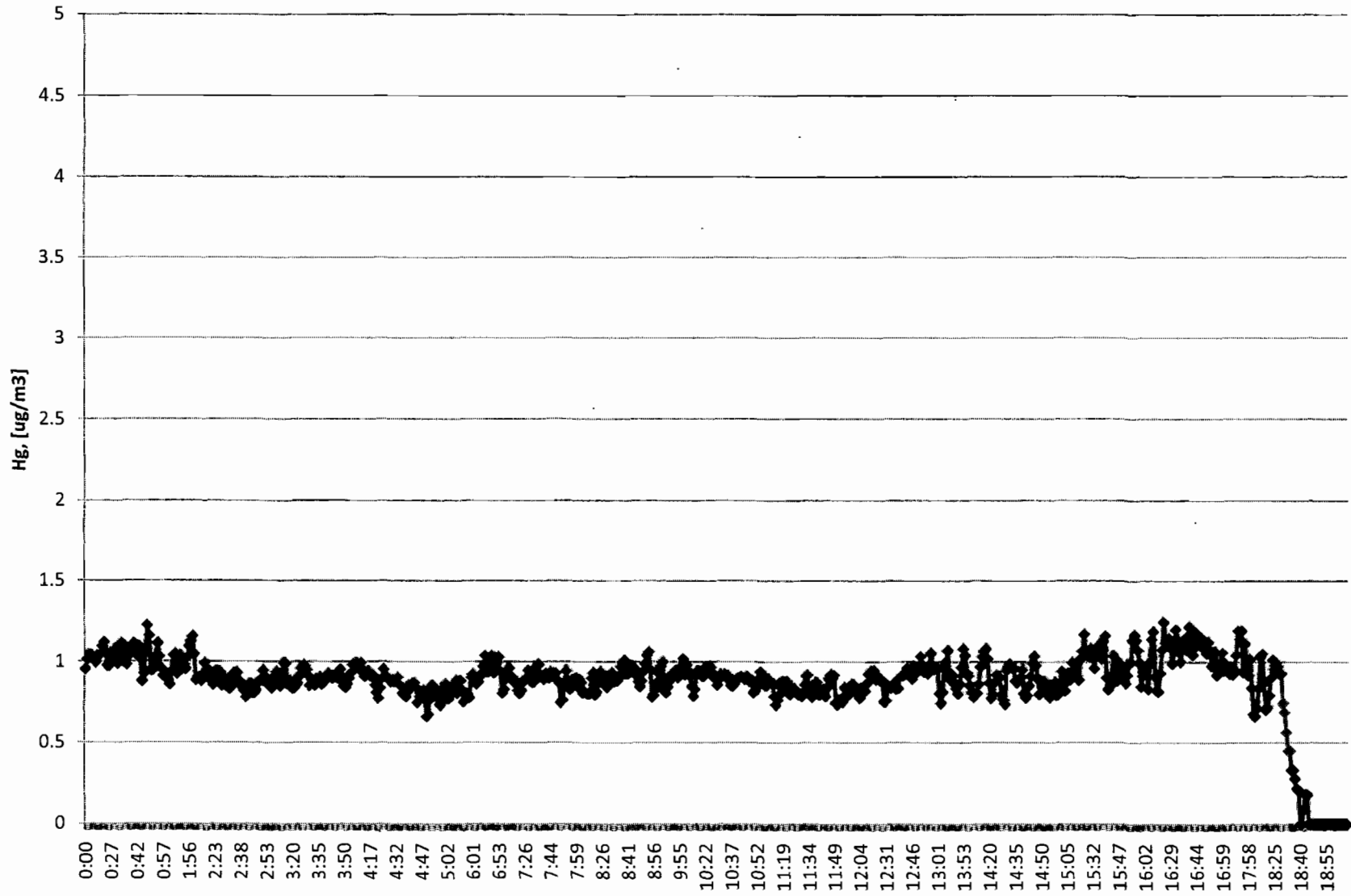
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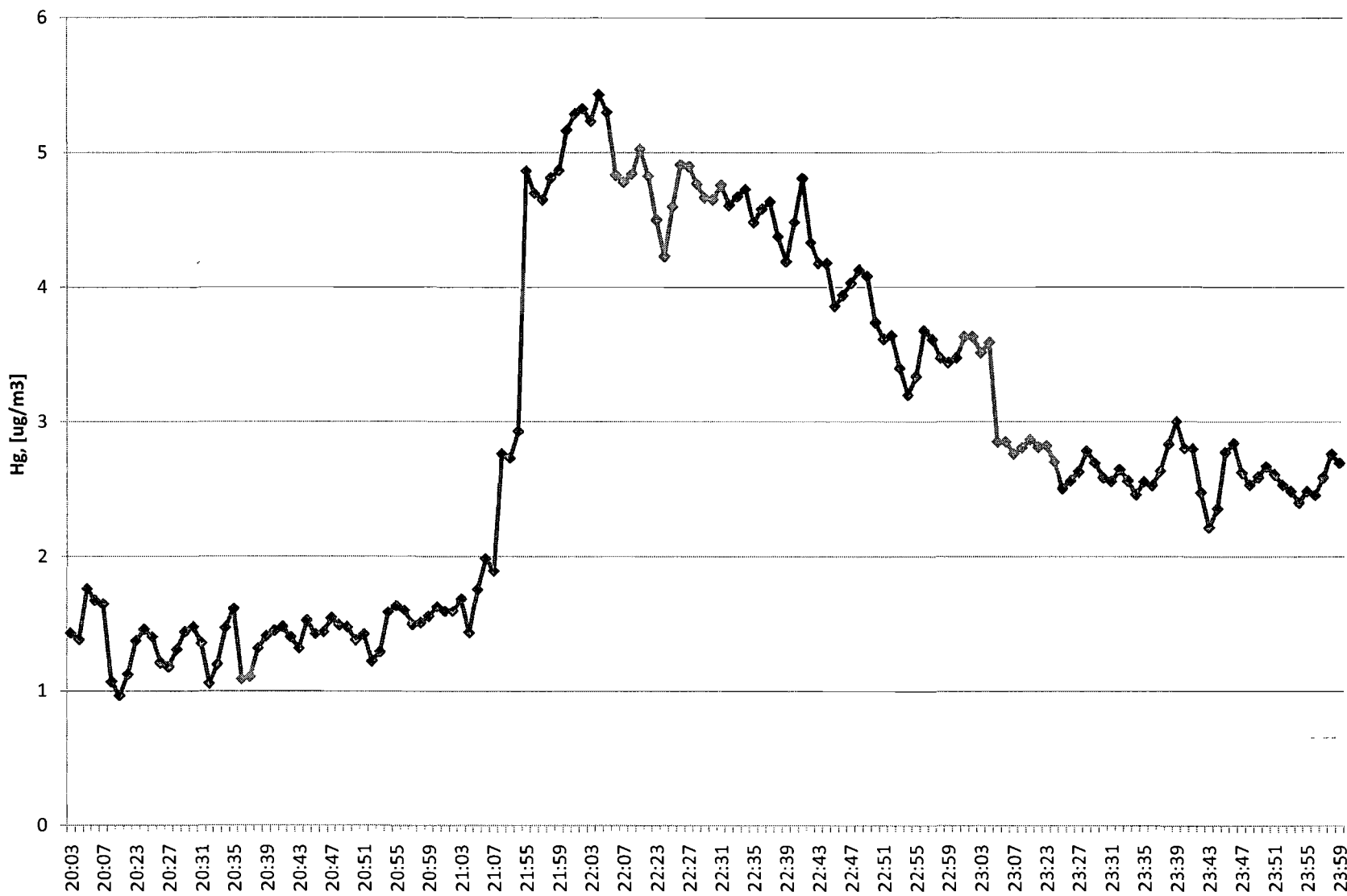
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10/31/2010



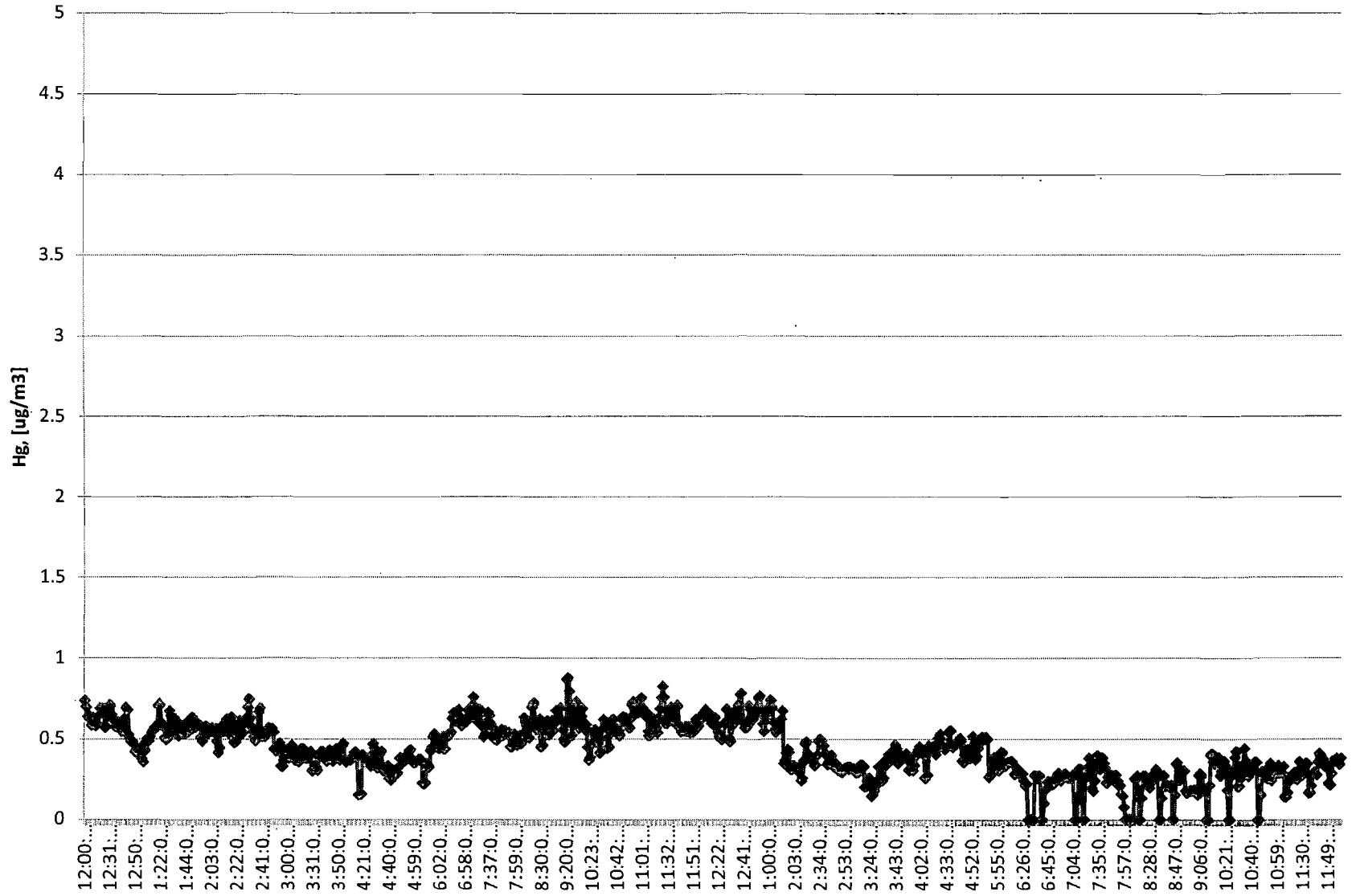
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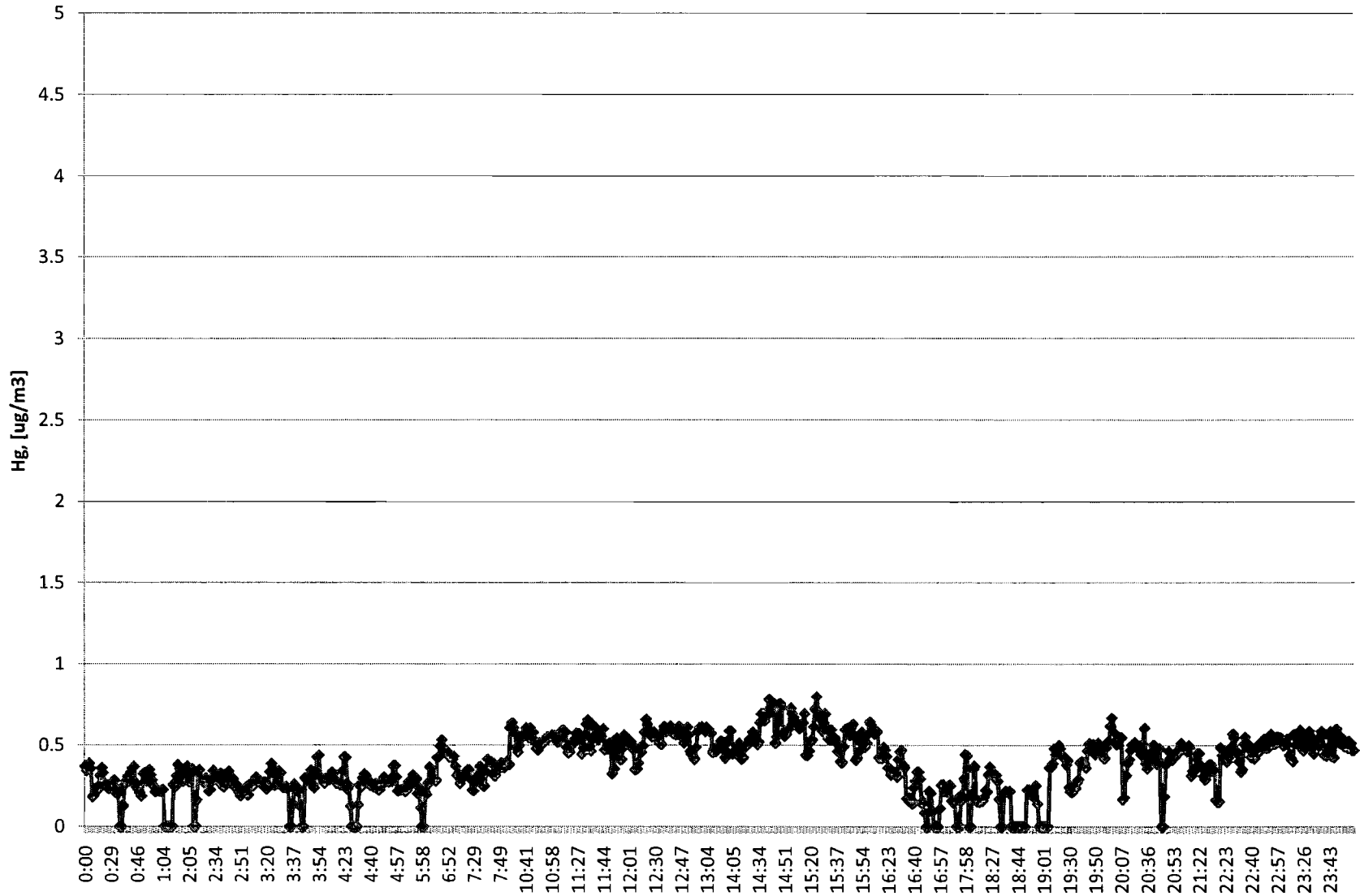


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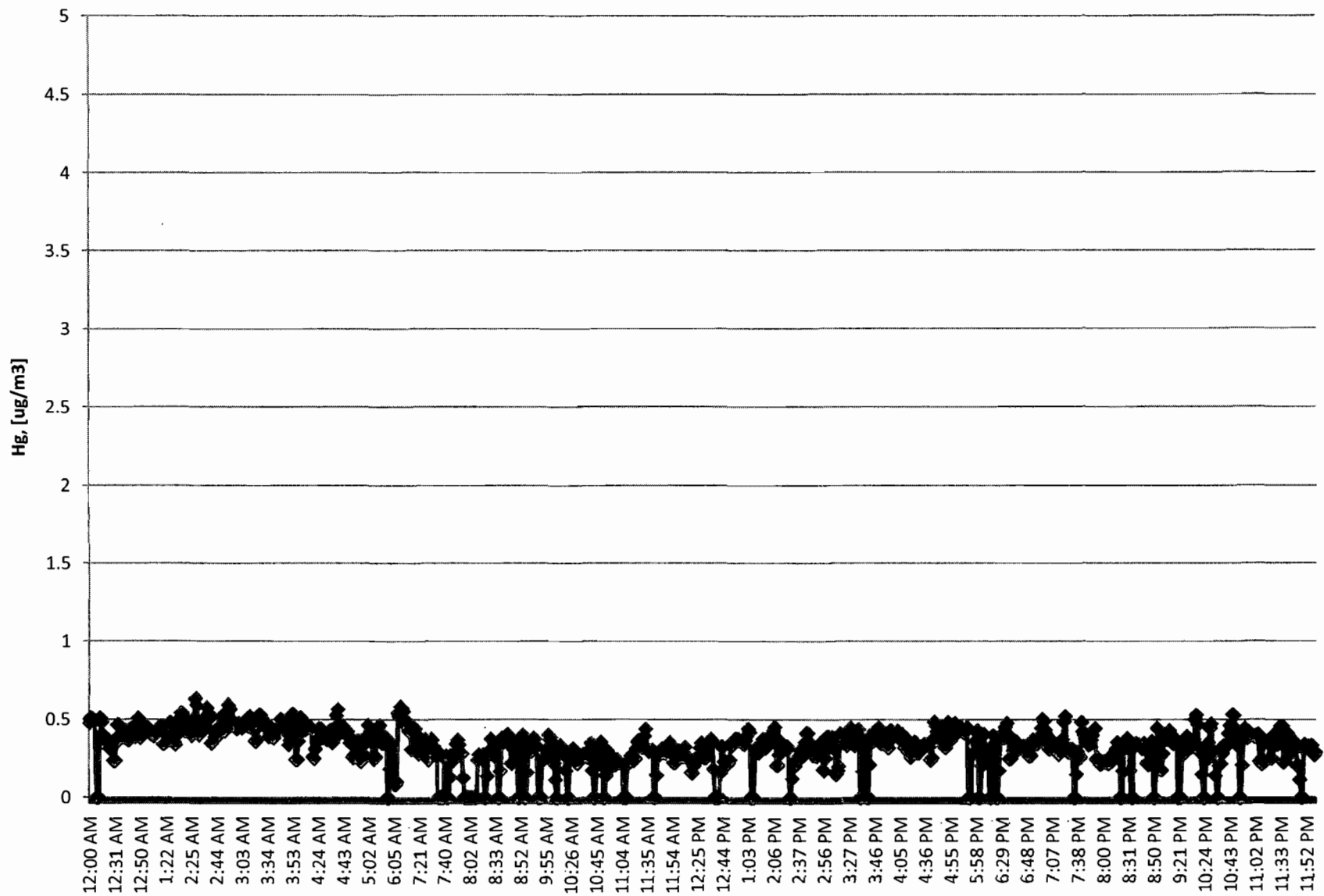




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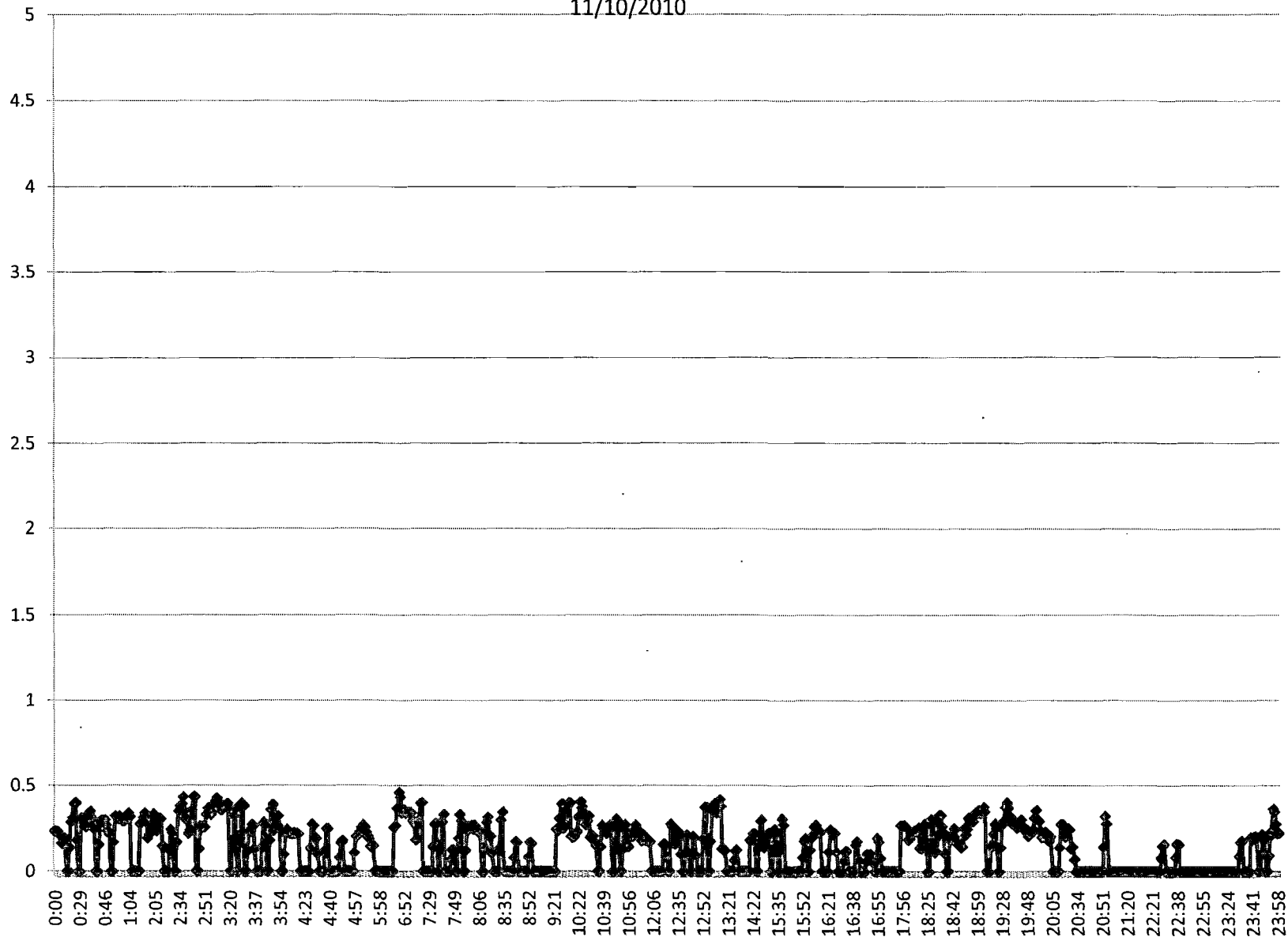


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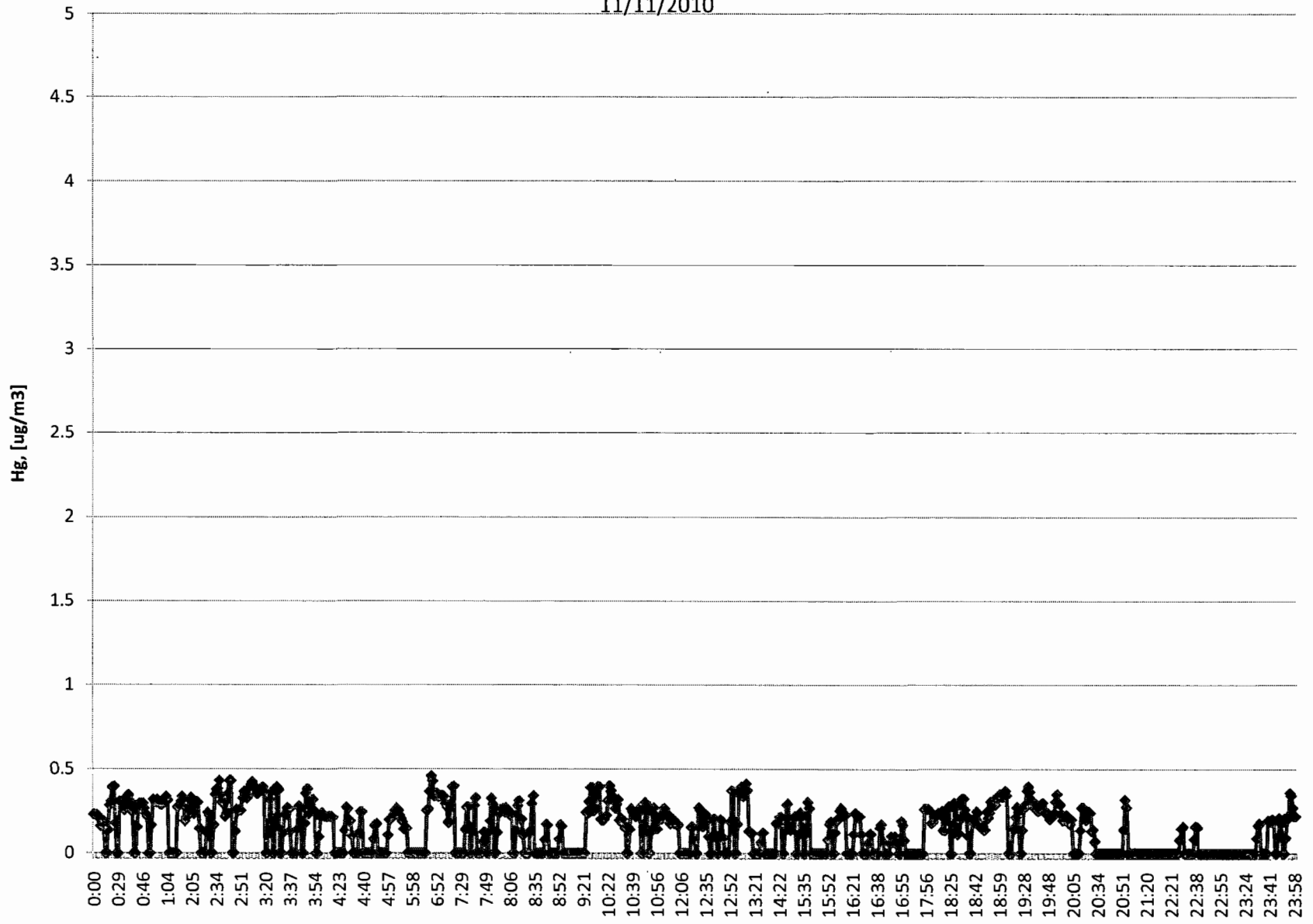


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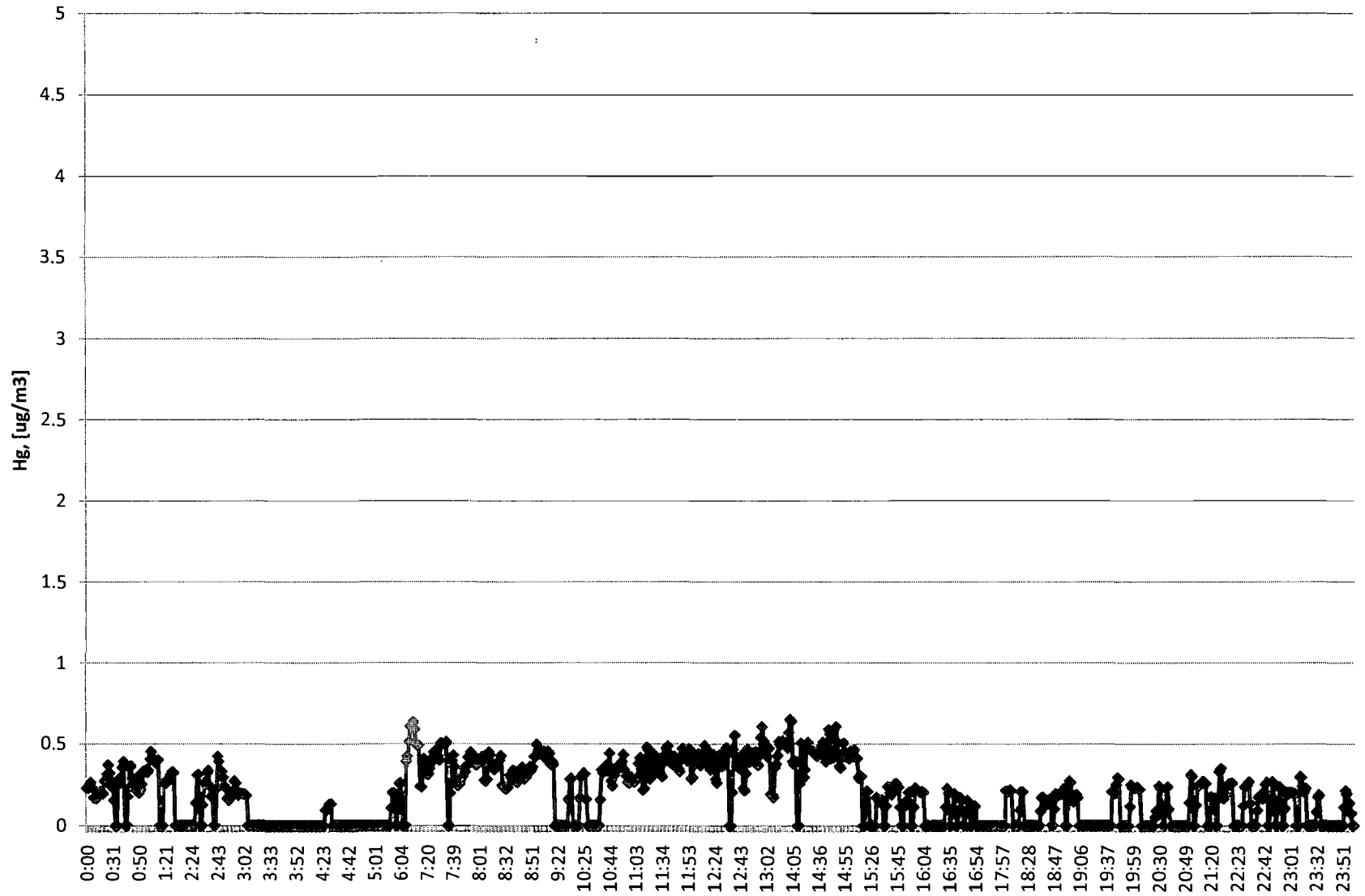
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11/11/2010



11/12/2010



11/13/2010

